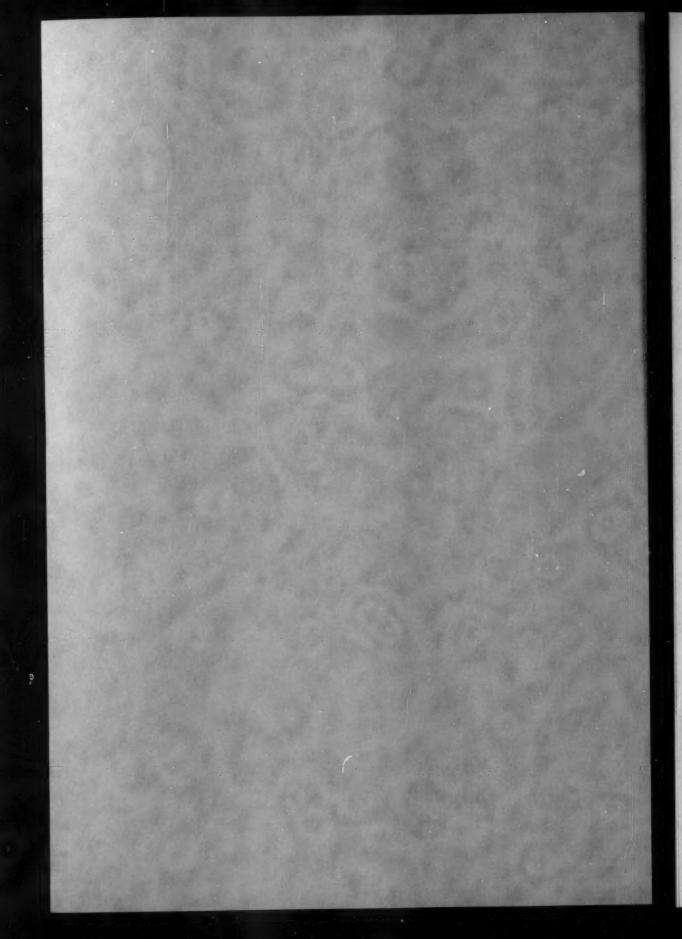
The Journal of the

SOCIETY OF DYERS AND COLOURISTS

Volume 72 Number 2

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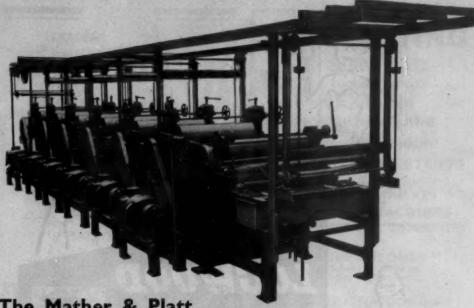
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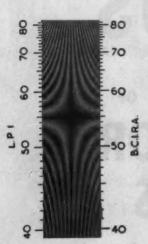
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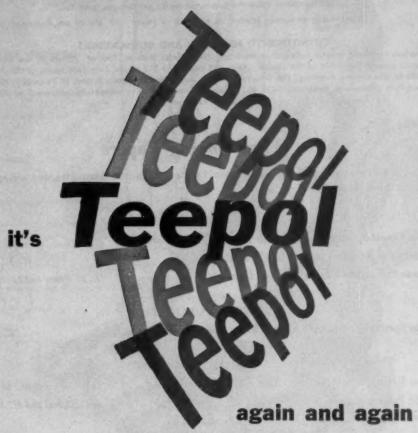
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The Journal of the Society of Dyers and Colourists

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NOTICE TO MEMBERS AND SUBSCRIBERS

Readers requiring general information regarding the Official Notices, List of Officers of the Society, etc. should consult pages 1-6 of the January 1956 and pages 369-373 of the July 1955 issues of the Journal, or write to The General Secretary, The Society of Dyers and Colourists, Dean House, 19 Piccadilly, Bradford 1, Yorkshire (Telephone Bradford 25138-9). Editorial Communications should be addressed to The Editor, at the same address.

Forthcoming Papers

The following papers have been accepted by the Publications Committee, and will appear in future issues of the Journal —

LECTURES

The Classification of Dyes by their Dyeing Characteristics Control of Reducing Agents in Vat Dyebaths

W. J. Marshall

Some Properties of Anthraquinone Dyes

R. H. Peters and H. H. Sumner

F.T.C.C. PUBLICATION

The Spectral Regions of Daylight which cause Fading

K. McLaren

W. Beal

COMMUNICATIONS

The Rotproofing of Viscose Rayon-II

J. W. Bell and M. M. Ramsey

The Polarography of Azo Dyes

Polarographic Contribution to an Investigation of

Fur Dyes and their Oxidation Products

J. de O. Cabral and H. A. Turner

G. Sandberg

THE SOCIETY OF DYERS AND COLOURISTS

ASSOCIATESHIP EXAMINATIONS

The next Associateship Examinations will be held on Thursday, Friday and Saturday, 14, 15 and 16 June 1956.

Closing date for receipt of candidates' applications 31 March 1956

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FORTHCOMING MEETINGS OF THE SOCIETY

Thursday, 23rd February 1956

MIDLAND'S SECTION. The Finishing of 'Terylene' and 'Terylene' Wool Blends. I. B. Haden, Esq., B.Sc., and B. I. Brooke, Esq., B.A. (Oxon.). Gas Theatre, Nottingham. 7 p.m.

NORTHERN IRELAND SECTION. Film Evening. Thompson's Restaurant, Donegall Place, Belfast. (Joint Meeting with the Textile Institute.) 7.30 p.m.

WEST RIDING SECTION. Progress in the Cibalan Dye Range and their Application. Clayton Dyestuffs Co. Ltd. Lecturer details later. The Victorial Hotel, Bridge Street, Bradford. 7.30 p.m.

Friday, 2nd March 1956

London Section. Basic Mechanisms of Fading of Dyed Textiles. H. B. Mann, Esq. (Courtaulds Ltd., Bocking). Royal Society Rooms, Burlington House, Piccadilly, London, W.1. 6 p.m.

Bradford Junior Branch. Annual Dance, Connaught Rooms, Bradford.

Tuesday, 6th March 1956

LEEDS JUNIOR BRANCH. The Textile Technologist in the Hosiery Trade. F. M. Stevenson, Esq., M.Sc., J.P. (Messrs. Stevensons (Dyers) Ltd., Ambergate, Derbyshire). The Lecture Theatre, Department of Colour Chemistry and Dyeing, The University, Leeds 2. 3.30 p.m.

Thursday, 8th March 1956

WEST RIDING SECTION. A Commentary on the Use of Colour in Wool. S. Morse-Brown, Bsq. International Wool Secretariat. Ladies to be invited. The Victoria Hotel, Bridge Street, **Bradford.** 7.30 p.m.

Friday, 9th March 1956

MIDLANDS SECTION. Midlands Section Dinner. Bull's Head Hotel, Loughborough.

Tuesday, 13th March 1956

SCOTTISH SECTION. The Soiling of Synthetic Fibres. G. G. Taylor, Esq., B.Sc., A.Inst.P. (Clayton Dyestuffs Co. Ltd.). St. Enoch's Hotel, Glasgow. 7 p.m.

BRADFORD JUNIOR BRANCH. Some Aspects of Continuous Vat Dying. Dr. R. J. Rallings (British Cotton Industries Research Association). Technical College, Bradford. 7.15 p.m.

Wednesday, 14th March 1956

NORTHERN IRELAND SECTION. Various Methods of Stripping Vat, Azoic, Direct and Sulphur Dyes. J. E. Riordan, Bsq. (College of Technology, Belfast.) Effect of pH on the Activity of Bleaching Agents. J. Boyd, Bsq. (College of Technology, Belfast). Thompson's Restaurant, Donegall Place, Belfast. 7.30 p.m.

Friday, 16th March 1956

MANCHESTER SECTION. Subject to be announced later. Textile Institute, 10 Blackfriars Street, Manchester 10, 6.30 p.m. Tuesday, 20th March 1956

HUDDERSFIELD SECTION. A New Approach to High Temperature Dyeing. S. N. Bradshaw, Esq. (Courtaulds Ltd.). Huddersfield. 7.30 p.m. (Venue later)

Wednesday, 21st March 1956

MIDLANDS SECTION. Fast Dyes on Cellulose Triacetate. J. G. Graham, Esq., B.Sc. (Joint Meeting with British Association of Chemists). Midlands Hotel, Derby. 7 p.m.

Thursday, 22nd March 1956

MIDLANDS SECTION. Dyeing Faults in the Carpet and Hosiery Industries. G. H. Lister, Esq., B.Sc., Ph.D. (Joint Meeting with the Textile Institute). Carpet Trades Canteen, Kidderminster. 7 p.m.

WEST RIDING SECTION. Annual General Meeting. The Victoria Hotel, Bridge Street, Bradford. 7.30 p.m.

Friday, 23rd March 1956

LONDON SECTION. Dyeing of Narrow Fabrics—Cellulosic and New Fibres. Dr. 1. M. S. Walls (I.C.I. Ltd.). George Hotel, Luton. 7 p.m.

Saturday, 24th March 1956

NORTHERN IRELAND SECTION. Annual General Meeting and Dinner. Details later.

Tuesday, 10th April 1956

Scottish Section. Textile Invention and the Dry Cleaner.
A. J. Crockatt, Esq., M.Sc., F.R.I.C. (Eastmans Dyers and Cleaners Ltd.). St. Enoch's Hotel, Glasgow. 7p.m.

Friday, 20th April 1956

Manchester Section. The Mechanism of Jigger Dyeing. Dr. Ashworth. Textile Institute, 10 Blackfriars Street, Manchester 10. 6.30 p.m.

LONDON SECTION. Fourth London Lecture. Colour in Fashion. James Laver Esq. (Keeper of the Departments of Textiles and Engraving, Illustrations and Design, The Victoria and Albert Museum. (Waldorf Hotel, London, W.C.2. 7 p.m. To be followed by a Dinner.

Saturday, 21st April 1956

Bradford Junior Branch. Annual General Meeting. Technical College, Bradford. 10.15 a.m.

Wednesday, 25th April 1956

HUDDERSFIELD SECTION. Visit to Messrs. Stevensons Dyers Ltd., Ambergate, Derbyshire.

MIDLANDS SECTION. A New Approach to High Temperature Dyeing. S. N. Bradshaw, Esq. Kings Head Hotel, Loughborough. Preceded by the Annual General Meeting of the Section. 6.30 p.m.

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The Centenary will be celebrated by Lectures and Social Functions in London on

Monday 7th May

Evening Principal overseas guests will be entertained by representatives of the Councils of the sponsoring bodies at Tallow Chandlers' Hall

in the City of London, prior to the opening of the Conference

Tuesday 8th May

Afternoon Opening paper on The Life and Work of Perkin will be read by Prof. John Read Ph.D M.A F.R.S

A Reception for all those attending Evening the Conference, and guests, at Guildhall in the City of London by kind permission of The Rt. Hon. The Lord Mayor of London

Wednesday 9th May Morning A paper

A paper on The Development of the Dyestuffs Industry will be read by Clifford Paine Esq n.sc

A paper on The Tinctorial Arts Today will be read by J. G. Evans Esq M.Sc.Tech Afternoon

A Banquet will be held at The Dorchester, Park Lane, London Evening

Thursday 10th May

Evening

A paper on The Development of Organic Chemistry since Perkin's Discovery will be read by Sir Alexander R Todd M.A D.Phil D.Sc Morning

LL-D F.R.S A Perkin Dinner and Ball will be given by the London Section of the Society of Dyers and Colourists at The Waldorf Hotel, Aldwych, London (Tickets 45s each)

All the papers will be read in the theatre of The Royal Institution, Albemarle Street, London

The charges for those attending the Conference and other functions are Conference Registration Fee and Guildhall Reception £3 3 0, Banquet £3 3 0

There is available an inclusive charge of £5 10 0 for Registration, Reception and Banquet but the charge for an accompanying guest for the Banquet is £3 3 0 and for the Guildhall Reception £1 1 0

Applications indicating desired ticket requirements for Bunquet, Receptions and Lectures should be sent AT ONCE to the General Secretary of the Centenary and please state whether you will be accompanied by a lady when attending the Banquet and Receptions

THE JOURNAL

OF THE

Society of Dyers and Colourists

Volume 72 Number 2

FEBRUARY 1956

Issued Monthly

COMMUNICATIONS

The Dyeing of Cellulose Acetate with Non-ionic Dyes III—Dyeing from the Vapour Phase

T. G. MAJURY

A study has been made of the absorption from the vapour phase, by secondary cellulose acetate, of five model dyes. These are p-nitroaniline, NN'-dimethyl-p-nitroaniline, azobenzene, p-aminoazobenzene. The amounts absorbed at equilibrium have been measured, and their dependence on the vapour pressure of the dyes and the temperature of the substrate has been established.

The vapour pressures of the dyes have been measured over a range of temperatures, those of azobenzene by manometry and the remainder by Knudsen's effusion method. The results together with the absorption data yield absorption isotherms from which are derived the isosteric heats of absorption of the dyes on cellulose acetate. The values, in the above order, are as follows— $-21\cdot2$, $-21\cdot4$, $-17\cdot8$, $-22\cdot0$, and $-22\cdot7$ kcal./mole.

The vapour pressure data are also used to evaluate the heats of vaporisation or sublimation of the dyes. These heats are applied to aqueous dyeing data already published to derive the absolute heats of association of the dyes on cellulose acetate. The results are in substantial agreement with the heats of absorption of the vaporised dyes.

A brief study has been made of the absorption by nylon and Terylene of vaporised azobenzene. The results for nylon yield a value for the heat of absorption of $-18\cdot0$ kcal./mole. The results for Terylene are insufficiently complete for quantitative treatment.

Introduction

The measurement and the characterisation of the forces of interaction between dye molecules and substrate form one of the topics at present under investigation. In a previous paper 1 it was suggested that the strength of these forces might be expressed as an "absolute" heat of association, defined as the difference in heat content of one mole of dye (a) in the vapour state and (b) dyed on the substrate. The vapour state was chosen for reference because it is the only one in which individual dye molecules do not interact physically with other molecules, e.g. those of the solvent, or other dye molecules as in the solid dye. With a group of non-ionic "model" dyes applied from aqueous solution to cellulose acetate, it was shown that the absolute heats of association may be evaluated by combining the heats of dyeing, of solution, and of sublimation of the dyes. It was necessary to assume that the water acts merely as a carrier of dye, exerting no permanent influence on the energy relations of the system.

It will be seen that the heat of association derived in this manner should be equal to the heat of dyeing from vaporised dye, i.e. to the heat which would be evolved if one mole of dye in the vapour state were transferred directly to the cellulose acetate. Measurement of this quantity would give

the strength of the dye-substrate bond directly; at the same time it would provide a check on the bond-strengths derived from the aqueous dyeing experiments and on the assumptions referred to above regarding the part played by water. The present work was carried out with these ends in view. It deals with the absorption from the vapour phase, by secondary cellulose acetate, of the five model dyes already used in aqueous dyeing, viz. p-nitroaniline, NN'-dimethyl-p-nitroaniline, azobenzene, p-aminoazobenzene. Some results are included on the vapour-phase absorption of azobenzene by 6,6-nylon and Terylene.

The data previously presented were incomplete in that published heats of sublimation were available for only two of the five dyes. The present work includes the measurement of the heats of sublimation of four of the dyes and the heat of vaporisation of the fifth, so that all the absolute heats of association can now be evaluated.

Experimental and Results

MATERIALS

The cellulose acetate was a commercial secondary product of 53.0% acetic acid content, prepared in the form of film by casting on a glass plate from acetone solution. The film thickness was 0.0015—

0.0017 cm. The nylon and the Terylene were undelustred fibre of 3.2 and 2.3 denier respectively.

The dyes were B.D.H. laboratory reagents recrystallised from suitable solvents. They were used in vacuo at temperatures up to 160°c. Their stability under these conditions may be presumed from references in the literature: according to Beilstein azobenzene and p-aminoazobenzene boil without decomposition at 293° and above 360°c. respectively; Berliner and May² found p-nitro-aniline to show appreciable decomposition only at temperatures above 260°c.; NN'-dimethyl-p-nitro-aniline melts without decomposition at 163°c.

ABSORPTION MEASUREMENTS

A diagram of the apparatus used is shown in Fig. 1. It consists of an inverted U-tube, one limb of which contains the sample of film or yarn suspended from a glass tripod, while the other contains the powdered dye. The U-tube was evacuated to 10^{-4} mm. of mercury or less and sealed off at the constriction as indicated by the dotted line. The tube was then placed with one limb in each of a pair of adjoining oil-baths, controlled to $\pm~0.3^{\circ}\mathrm{C}$, the limb containing the film being at a higher temperature than the limb

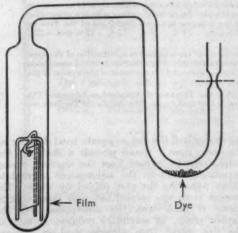


Fig. 1- Absorption Apparatus

containing the dye. Under these conditions the film was surrounded by unsaturated dye vapour, so that dye could not condense on the film. The horizontal tube was wound with resistance tape and heated by passing a current sufficient to prevent the deposition of dye in the tube; pre-liminary tests with a thermocouple were made to ensure against excessive heating of the tube.

To find the amount of dye absorbed by the sample, the tube was removed from the oil-baths and opened instantly so as to avoid a shift of equilibrium on cooling. The dye was then brought into solution and estimated photometrically by means of a Uvispek (Hilger & Watts) spectrophotometer. Cellulose acetate film was dissolved in a mixture of methylene chloride and ethanol in the ratio 3: 2 by volume. Nylon and Terylene could be dissolved similarly in a mixture of phenol

and chloroform in the ratio 3:2 by weight, but it was found preferable to avoid the use of this or other troublesome solvents by desorbing the dye and then dissolving it in the methylene chloride-alcohol mixture. The desorption was carried out very simply by sealing off a weighed sample of fibre in a straight evacuated tube, which was then held vertically overnight with the lower portion, containing the sample, in an oil-bath at 150°c. The desorbed dye crystallised in the cool upper portion of the tube. The results were concordant with those given by the solution method.

A small number of experiments were carried out with saturated dye vapour, i.e. with the film and the condensed dye phase at the same temperature. For these the film was suspended over the dye in a test-tube, which was evacuated, sealed off, and heated in an oven at the required temperature

(see Table I).

In all cases the absorption of dye took place uniformly and, when equilibrium was reached, with complete penetration of the film or fibre, as was shown by microscopic examination of sections. No surface deposits of dye were observed even in saturated dye vapour. With p-nitroaniline and p-aminoazobenzene, however, saturated or nearly saturated vapour, particularly at higher temperatures, caused marked swelling and shrinking of the acetate film. This was accompanied by anomalously high degrees of absorption. These results are marked by an asterisk in the tables and are not included in the absorption isotherms.

For p-nitroaniline and azobenzene, the first two dyes studied, the experiments were performed in duplicate. The reproducibility was found to be within 3%, and most subsequent experiments

were performed singly.

The attainment of equilibrium was ensured by increasing the duration of the experiment until no further increase in absorption was observed. In general, runs were carried out over periods varying from overnight to several days, the times required being greater the lower the temperature of the substrate and the larger the molecules of dye. Convenient rates of absorption were obtained at temperatures in the range 120–160°c.

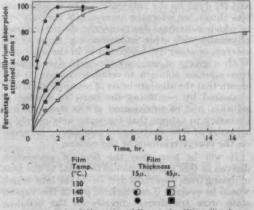


Fig. 2—Rate of Absorption of Vaporised p-Nitronniline by

TABLE I

Absorption of Vaporised Model Dyes by Secondary Cellulose Acetate

		Absorption of va	portseu Mi	der Dyes by Se			
		Film temp., °c	120°	130°	140°	150°	160°
1	Oye Temp. (°c.)	Vapour Pressure (Hg)		Amount of I	ye absorbed (g.	/100 g. film)	
			p	-NITROANILINE			
	100	21.5 μ.	8-16	4-48	2.41	1.32	-
	110	48-8	- 16.5 .	8-90	4.79	· 2-67	-
	120	106	50*	18-8	9-62	5-65	
	130	224			24-2*	10-4	
	140	453			-	29-9*	4000
			NN'-Dn	ETHYL-p-NITROAM	VILINE -		
	100	17-5 μ.	2-20	1.20	0.78	0-40	_
	105	26-7	3-22	1.80	1-12	0.61	-
	110	40-2	5-60	2-66	1.51	0.92	(aross)
	115	60-0	_	4.09	2-13	1.20	_
	120	88-1	_	5-70	3.11	1.71	-
	125	130			4-74	2-41	-
	130	189		_	6-42	3-44	-
	135	270			eren .	4.99	-
	140	385			_	7-18	even
				AZOBENZENE			
	100	0-73 mm.	5-20	3-04	1.97	1.21	_
	110	1.27	8-85	5.08	3-00	2.01	-
	120	2.12	14-7	8-55	5.00	3-02	_
	130	3-48		15-1	8-06	4.83	-
	140	5-56			12.7	7-48	_
	150	8-67				11-2	-
			p-A	MINOAZOBENZENE			
	100	7-15 д.	7-39	4-04	2.05	1-16	_
	105	11.5	11.8	5-68	3-17	1.85	-
	110	18-2	18-2	8-95	4.65	2-11	-
	115	28-5		13-1	6-67	3.73	
	120	44-0	-	18-0	9-32	5-36	-
	125	67.8		-	13-6	7-11	
			NN'-DIMET	HYL-p-AMINOAZOB	ENZENE		
	100	4-48 μ.	_	_	0.58	0-37	0.18
	105	7.52	-		-	-	0.29
	110	12-4	_		1.40	0.78	0.46
	115	20-2	-	-	2.20	1.22	0.74

Systematic study of the rate of absorption was not attempted, since the interest of the work was centred on equilibrium data; however, some rate curves were obtained for p-nitroaniline on cellulose acetate and are shown in Fig. 2. The ordinates give $100~M_t/M_{\odot}$, where M_t is the absorption at time t and M_{\odot} is the absorption finally attained under the conditions in question. So far, the data are insufficient to allow a quantitative interpretation of these curves, or a comparison of the rates of absorption with those found in aqueous dyeing 3 .

The results for cellulose acetate are presented in Table I, expressed as grams of dye absorbed per 100 g. of undyed, air-dry substrate. The first two columns in each section of the table give the temperatures and corresponding vapour pressures of the dye in question. The determination of these pressures is described in the next section.

The results obtained with nylon and Terylene will be dealt with separately at the end of the paper.

VAPOUR PRESSURE MEASUREMENTS

The vapour pressures of the model dyes were required for two purposes—(1) to enable the vapour-phase absorption data to be presented in the form of isotherms with a view to determining the heats of absorption, and (2) to give the heats of sublimation or vaporisation of the dyes; these would complete the data needed for the determination of the absolute heats of association from the aqueous dyeing work 1 and so lead directly to a comparison of the latter with the heats of absorption from the vapour phase.

The heats of sublimation L_{sub} or vaporisation L_{vap} were calculated from the vapour pressure data by means of the Clausius-Clapeyron equation—

$$L = T \frac{\mathrm{d}p}{\mathrm{d}T} (v - v') \tag{i}$$

where T is the absolute temperature, p the pressure, v the volume of one mole of vapour, and v' that of one mole of the condensed phase. Neglecting v', substituting RT/p for v, and integrating, we find that—

$$\ln p = \text{Constant} - \frac{L}{RT} \tag{ii}$$

where R is the gas constant, 1.986 cal./mole per °c. The experimental values for pressure were plotted logarithmically against 103/T, giving straight lines of the form-

$$\log_{10} p = a + \frac{10^9 b}{T} \tag{iii}$$

Values of a and b and their probable errors were calculated by the method of least squares, and L was obtained from the relation-

$$L = -2.303 Rb \text{ keal./mole}$$
 (iv)

The vapour pressure of azobenzene was measured over the range 90-140°c. by means of a simple U-tube mercury manometer of 5 mm. bore. The azobenzene was distilled under vacuum into a bulb attached to the manometer, and degassed by melting under vacuum. The manometer and bulb were sealed off, and immersed in an oil thermostat controlled to ± 0.1°c. The difference in mercury levels was measured by means of a telescope to 0.01 mm., five concordant readings being taken at each temperature. The averaged readings are given in Table II, and the corresponding Clausius-Clapeyron plot is shown in Fig. 3. The values of a, b, and $L_{\rm vap}$ are given in Table III.

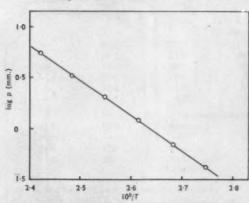


Fig. 3- Vapour Pressure p of Azobenzene

TABLE II Vapour Pressure of Azobenzene

Temperature, °c. 100.0 109-6 129-8 140-0 Vapour pressure, 0.44 0.70 1.20 2.05 3.32 5.53

The vapour pressures of the remaining four dyes, being too low for the use of a manometer, were obtained by a method, due to Knudsen 4, in which the rate of effusion of vapour through a hole of known size is measured. The apparatus, consisting essentially of an effusion vessel suspended from a spiral spring balance, was similar to that described by Bradley and Care 5, except that a glass instead of a silica spring was used. The spring was 0.29 mm. thick and 2 cm. in diameter. It was annealed by heating for several days at 200-210°c. while under its subsequent working

load. The sensitivity was 0.329 mm./mg. This was determined before and after the series of vapour pressure measurements, and was found to have remained unchanged.

The effusion vessel consisted of a bulb of soft glass about 1 cm. in diameter fitted with a window" of platinum foil 0.002 in. (0.051 mm.) thick in which a small hole had been carefully bored. The size of this hole limits the maximum pressure for which the cell is suitable in two ways. Firstly, the mean free path of the vaporised molecules in the vessel should always be large compared with the diameter of the hole; and secondly, the rate of effusion should be sufficiently low to avoid self-cooling of the material under observation. Thus if the pressure is increased beyond the limits imposed by the size of the hole, the corresponding increase in the rate of effusion will be too low. To eliminate these errors two cells were made with holes of diameter (A) 0.05 in. and (B) 0.025 in. and were used only in regions of pressure in which collinear results were obtained. The cells were calibrated by measuring the rate of effusion of benzophenone, which has been subjected to careful measurement by Neumann and Völker 6. The benzophenone was distilled in a high vacuum at 140°c.; the distillate, in the unstable liquid form, was converted to the stable solid form by adding a small crystal of the latter. The calibration showed that cell A was suitable for pressures up to 5μ , and cell B for pressures up to $10 \,\mu$. of mercury.

The recrystallised dyes were repurified for the effusion measurements by sublimation in a high vacuum with continuous pumping. For each dye the temperature range which gave the range of pressures suitable for each of the two cells was ascertained and rates of effusion were measured at intervals within this range. Then for equal rates of effusion of benzophenone and of dye the vapour pressure of the dye was calculated from the effusion equation 4

effusion equation
$$\frac{w}{k} \times \left(\frac{2\pi RT}{M}\right)^{\frac{1}{4}}$$
 (v) whence—
$$p_{0} = p_{0} \times \left(\frac{M_{b}}{M} \times \frac{T_{0}}{\sigma}\right)^{\frac{1}{4}}$$
 (vi)

$$p_{\rm u} = p_{
m b} imes \left(rac{M_{
m b}}{M_{
m u}} imes rac{T_{
m u}}{T_{
m b}}
ight)^{rac{1}{2}}$$
 (vi

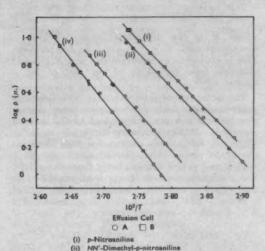
where p_u , M_u , and T_u are the vapour pressure, molecular weight, and absolute temperature of the unknown, and p_b , M_b , and T_b are those of benzophenone; w is the weight of material effusing in unit time; and k is the cell constant. It is assumed that the effusing species is monomolecular; evidence on this point will be given below. The results are plotted in Fig. 4.

It will be seen that the plots of Fig. 3 and 4 are accurately linear; this linearity justifies the use of the Clausius-Clapeyron equation and the implicit assumption of the constancy of L over the temperature range of the measurements. The values of a, b, and L are given in Table III.

Previously published data are available only for azobenzene and p-nitroaniline. For azobenzene, Anschütz and Reitter? gave b.p. of 293°c. at normal pressure and 155°c. at approx. 12 mm.

TABLE III

Vanoue Pressures and	Heats of Vaporisation	or Sublimation of Model Dye	200
Vanour Pressures and	TICHER OF AMDOLISHMENT	or addition or wroter by	-



(iv) NN'-Dimethyl-p-aminoazobenzene
Fig. 4— Vapour Pressures by the Effusion Method

a-Aminoszobenzene

(iii)

pressure; these values correspond to an approximate heat of vaporisation of 14.5 kcal./mole. Extrapolation of the present data gives the b.p. at 760 mm. as 286 ± 16°c. For p-nitroaniline, Wolf and Trieschmann 8 and Wolf and Weghofer gave heats of sublimation of 24.7 ± 0.4 and 23.6 ± 0.6 kcal./mole respectively; no actual pressures were quoted. Berliner and May 2 measured the vapour pressure of liquid p-nitroaniline in the range 190-250°c. and found the heat of vaporisation to be 18.5 kcal./mole. The heat of fusion of p-nitroaniline has been found to be 5.0 kcal./mole 10. Combining these two values gives 23.5 kcal./mole for the heat of sublimation. The agreement between these results and the present ones is satisfactory.

Comparison of the temperature ranges over which the pressure measurements were made with those over which absorption data were obtained (Table I) shows that for the two nitroanilines considerable extrapolation of the pressure measurements is needed. (Direct measurement by effusion or manometry in the range 100–140°c. was impracticable owing to the limitations of the methods.) The low degree of scatter in the effusion data enables this to be done with sufficient accuracy

provided that no change in heat of sublimation occurs at temperatures outside the measured range. Such a change might be caused by a change of state in either the solid or the vapour phase, e.g. a change in the crystalline form of the dye or a change from associated to monomolecular vapour. As a check on this possibility the vapour pressure data of Berliner and May $^{\circ}$ were extrapolated down to the melting point of p-nitroaniline, giving a pressure of 0.92 mm. at 148°c. The present data extrapolated up to the melting point gave 0.77 \pm 0.25 mm. As a direct experimental check the vapour pressure of a twice sublimed sample of p-nitroaniline was measured by a mercury manometer at 148°c.; the pressure was 0.73 mm.

A similar manometric measurement on NN'-dimethyl-p-nitroaniline gave $1\cdot 1$ mm. at $160^{\circ}\mathrm{c}$, the value calculated from the effusion data being $1\cdot 5\,\pm\,0\cdot 7$ mm.

This agreement supports the validity of the extrapolation of the effusion data. Considering also the wide range of temperatures over which linear results are found, viz. 70–250°c. for p-nitroaniline, 70–160°c. for NN'-dimethyl-p-nitroaniline, and 90–293°c. for azobenzene, there appears to be no likelihood of association in the vapour phase.

The low vapour pressures of the two aminoazobenzenes coupled with their low melting points made a manometric check impracticable, but the extrapolations required here are in any case relatively small.

Results and Discussion

The absorption and vapour pressure data were combined in absorption isotherms, most of which are quite closely represented by straight lines passing through the origin; i.e. Henry's law is obeyed. Those for p-nitroaniline, NN'-dimethylp-nitroaniline, and azobenzene (Fig. 5 and 6) are linear within the experimental error, though there may be a small departure at the highest pressures in the isotherms at 150°c. for p-nitroaniline and azobenzene. For p-aminoazobenzene, only the isotherm at $120^{\circ}\mathrm{c.}$ is perfectly straight, and those at higher temperatures show a slight but definite curvature concave to the pressure axis. The experimental points for NN'-dimethyl-p-aminoazobenzene are best fitted by straight lines passing a little above the origin, so a slight curvature is implied in the low-pressure region, though no data are available here.

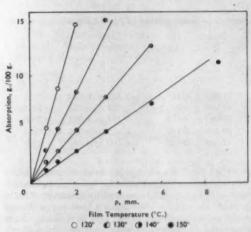


Fig. 5— Isotherms for Absorption of Vaporised Asobensens on Secondary Cellulose Acetate

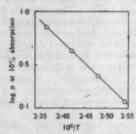


Fig. 6— Clausius-Clapeyron Plot for Absorption of Vaporised [Azobenzene on Secondary Cellulose Acetate (10 g./160 g.)

The fact that Henry's law is obeyed or only slightly departed from is particularly striking in view of the substantial amounts of absorption which occur; it is comparable with the constant partition of dyes between substrate and water observed in the aqueous dyeing of cellulose acetate and nitrate, nylon, and Terylene (see Part I¹ for references). Here, as in aqueous dyeing, it appears that there is no question of absorption on a limited number of sites. Indeed, considering the wide range of adherence to Henry's law, it may be better to think not of specific absorption sites but rather of a solvent-solute relationship in which

bonds are continuously broken and reformed as a result of the thermal motion of both components. On this view the curvature observed in some of the isotherms can be regarded as evidence of variation in the activity coefficient of the dye in the cellulose acetate phase.

The heats of absorption of the vaporised dyes were evaluated by applying the Clausius-Clapeyron equation in a similar manner to that already used to evaluate the heats of sublimation. For a fixed amount of dye absorbed, the pressure of vapour in equilibrium with the substrate is given by the equation—

$$\ln p = \text{Constant} + \frac{H}{RT}$$
 (vii)

where H is the differential (isosteric) heat of absorption and T is the absolute temperature. This is analogous to equation (ii), and H is again obtained from the slope of the plot of $\log p$ against $10^3/T$.

The isotherms of p-nitroaniline, NN'-dimethylp-nitroaniline, and azobenzene, being linear, give heats which are independent of the amount absorbed. Arbitrary absorption levels at 10%, 5%, and 10% were chosen for the Clausius-Clapeyron plots, which yielded heats of -21.2, -21.4, and -17.8 kcal./mole for these three dyes. For p-aminoazobenzene heats were evaluated at $2\%, \, 4\%, \, 8\%, \, \text{and} \, 14\% \, \text{absorption, giving} \, -22 \cdot 0, \, -23 \cdot 5, \, -26 \cdot 0, \, \, \text{and} \, \, -26 \cdot 0 \, \, \, \text{kcal./mole.} \, \, \text{For}$ NN'-dimethyl-p-aminoazobenzene the heat of absorption, evaluated at 0.5% absorption, was 22.7 kcal./mole; the straight portions of the isotherms at higher absorptions yielded values which did not differ significantly from this, and no use could be made of the curved portions at lower absorptions, since these were not supported by experimental data.

These heats of absorption are collected in the last column of Table IV. Their probable error is the sum of the errors arising in the vapour pressure measurements and in the absorption measurements and is estimated to be approx. ± 1 kcal./mole.

The second column of Table IV gives the heats of condensation of the dyes, i.e. the heats of sublimation or vaporisation with reversed sign, which can be compared with the heats of absorption. It will be seen that for the first three dyes,

TABLE IV

Absolute Heats of Association and Heats of Absorption from the Vapour Phase on Secondary
Cellulose Acetate (kcal./mole)

	Centillose Heetil	(2000)		
Dye	Heat of Dyeing + Heat of Soln.	Heat of Condensation to Solid (8) or Liquid (L)	Absolute Heat of Association	Heat of Absorption from Vapour Phase
p-Nitroaniline	. 2.9	-23·3(S)	-20.4	-21.2
NN'-Dimethyl-p-nitroaniline	3-8	-23-6(8)	-19-8	-21.4
Azobenzene	-1.2	-15·5(L) -17·7(S)	-16-7	-17-8
p-Aminoazobenzene	1.2	-26·5(S)	-25-3	-22.0
NN'-Dimethyl-p-aminoazobenze	ne 0-8	-28.9(8)	-28-1	-22.7

whose heats of absorption are independent of the amount absorbed, the heats of condensation to the solid dye are almost equal, within experimental error, to the heats of absorption. For p-aminoazobenzene the heat of absorption is significantly lower than the heat of condensation for small amounts absorbed, then rises to virtual equality at 8% absorption, and remains constant at this level for higher degrees of absorption. Presumably the lower value of -22.0 kcal./mole is characteristic of the absorption of p-aminoazobenzene on cellulose acetate, but with increasing absorption the substrate approaches the character of p-aminoazobenzene and thereby acquires increased ability to bond freshly deposited molecules of the latter. Increasing heats of sorption with increasing amounts of vapour sorbed are characteristic of systems in which the heat of absorption is smaller than the heat of condensation 12; more often this relation is reversed and the heat of sorption falls as absorption proceeds, as in the case of cellulose For NN'-dimethyl-p-aminoazoand water 13. benzene the heat of absorption is again lower than the heat of condensation, but shows no tendency to rise, possibly because the amounts of dye absorbed are too small to change the absorbing properties of the substrate.

Before discussing the heats of absorption further it will be helpful to establish their relationship to the absolute heats of association derived from aqueous dyeing experiments.

COMPARISON WITH ABSOLUTE HEATS OF ASSOCIATION

Part I¹ reported the heats of solution and the heats of dyeing from aqueous solution of the five model dyes. Heats of sublimation were available only for p-nitroaniline and azobenzene. The data were used to evaluate the heats of association on cellulose acetate of these two compounds, while those of the other three were estimated roughly by inference from their polar and hydrogen-bonding properties. These results can now be presented in complete form with the aid of the vapour pressure data given in Table III.

For this purpose the aqueous dyeing experiments with azobenzene have been revised; the uncertainty due to the previous experiments being above or below the melting point has been removed by substituting results obtained at temperatures above the melting point only. Samples of secondary accetate film were dyed in saturated solutions of azobenzene at 70°, 80°, and 90°c. The amounts of azobenzene absorbed at equilibrium are given in Table V.

TABLE V

Absorption of Azobenzene from Saturated Aqueous Solutions by Secondary Cellulose Acetate

Temperature, °c. ... 70 80 90 Uptake, g./100 g. film 4·31, 4·38 4·15 4·05

Assuming as before that absorption at a given temperature is proportional to dye concentration, and absorption at a given dye concentration decreases exponentially with increasing temperature, it follows that the amount absorbed from a saturated solution is given by—

 $x = \text{Const.} \times e^{-(H+Q)/RT}$

where T is the absolute temperature, H is the heat of dyeing, and Q the heat of solution of azobenzene. A plot of $\log x$ against $10^3/T$ should be a straight line whose slope gives the sum of the heats of dyeing and solution. This plot is given in Fig. 7; the slope corresponds to $H+Q=-1\cdot 2$ kcal./mole. This differs only slightly from the value of $-0\cdot 6$ kcal./mole previously reported.

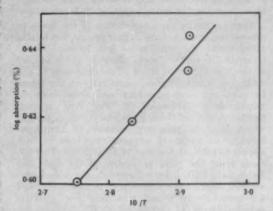


Fig. 7— Absorption of Ambensene from Saturated Aqueous Solution by Secondary Cellulose Acetata

The data now available are presented in Table IV. The values in the first column, with the exception of that for azobenzene, are the same as those in the fourth column of Table II in Part I¹, but with reversed sign. The probable error of the figures in the fourth column is estimated as before to be \pm 2 kcal. This error and the error of \pm 1 kcal. assigned to the heats of absorption are not quite independent, as both include \pm 0-4 kcal. from the heats of sublimation; however, it will be reasonable to disregard differences of less than 3 kcal./mole in comparing the two sets of values.

On this basis, the absolute heat of association is equal to the heat of absorption of vaporised dye for p-nitroaniline, NN'-dimethyl-p-nitroaniline, and azobenzene. For p-aminoazobenzene there is a difference, which is barely significant, between the absolute heat of association and the initial heat of absorption; if the comparison is made at the absorption level of the aqueous experiments (4-14%) the difference vanishes. For NN'dimethyl-p-aminoazobenzene the absolute heat of association appears to be significantly greater than the heat of absorption. No explanation can be offered at present for the apparent difference in the case of the last two compounds. It may arise from failure of the assumption that the heat of wetting of the substrate is unchanged by dyeing, but it is difficult to see why a change in the wetting behaviour should arise only with the aminoazobenzenes. More probably it is connected with non-ideal behaviour of these compounds in the cellulose acetate phase, for which evidence exists in the curvature of their vapour absorption isotherms.

We may conclude that the two methods of measurement of bond strength give closely comparable results for three of the five model dyes, while there is a suggestion that for the two amino-azobenzenes the aqueous dyeing method leads to slightly higher values than the vapour absorption method. The vapour method is undoubtedly to be preferred as a measure of bond strength on the grounds of directness and freedom from assumptions regarding the heat of wetting of the substrate.

Turning finally to consideration of the heats of absorption in relation to dye structure, the results substantiate the conclusions put forward tentatively in the earlier paper. Firstly, the heats are of the same order as those of condensation to the solid dye, which implies a similarity in the forces between dye and substrate and those between neighbouring molecules in the dye crystal. Secondly, the existence of primary amino groups, which may be presumed capable of hydrogenbonding to carbonyl oxygen, does not in fact appear to increase the heat of absorption. Thirdly, considering the polar properties of the dyes (see Table III, Part I for references), the two nitroanilines, which have large dipole moments (6.5 and 6.9 e.s.u. \times 10^{18}) and relatively low electron polarisabilities (41 and 56 c.c./mole), show similar heats of absorption to the two aminoazobenzenes, which have relatively low dipole moments (2.7 and

 $3.2 \text{ e.s.u.} \times 10^{18}$) and large polarisabilities (71 and 79 c.c./mole). Azobenzene, which has an intermediate polarisability (62 c.c./mole) but zero dipole moment, shows the lowest heat of absorption. These results are consistent with the view that polarisability and permanent polarity both play a part in the formation of bonds with cellulose acetate, the former giving rise to dispersion and induced-dipole interaction, the latter to dipoledipole interaction. Dispersion forces depend on the product of the polarisabilities of the interacting species, i.e. on the square of the polarisability in the case of self-interaction in the dye crystal. Since the aminoazobenzenes are highly conjugated and the substrate is unconjugated and hence of relatively low polarisability, this may explain why the heats of absorption of the aminoazobenzenes fall below their heats of condensation.

Nylon and Terylene

Some preliminary experiments have been carried out on the absorption of azobenzene vapour by nylon and Terylene. The results indicate behaviour similar to that found for cellulose acetate. Insufficient experiments were made with Terylene (Table VI) to allow more than this qualitative assessment, but with nylon enough data were obtained to give a reasonable indication of the absorption isotherms. These are illustrated in

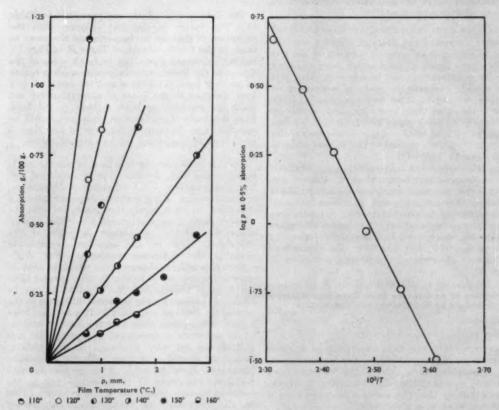


Fig. 8— Absorption of Azobensene Vapour on Nylon

TABLE VI

Absorption of Azobenzene from the Vapour Phase by Terylene

(g./100 g. air-dry, undyed fibre)

Dye Temp.	p (mm.)	Temp. 140°	of Terylene 150°	(°c.) 160°
100	0.73	0.66	0.39	0-27
105	0.97	0.65	-	0.28
110	1.27	-	0.66	0-34
115	1.64	Line !	0.73	0-58
120	2.12	-	-	-
125	2.72	-	-	-
135	4-41	-	1.92	-
140	. 5-56	-	2.68	-

Fig. 6 together with the corresponding Clausius-Clapeyron plot. The heat of absorption of azobenzene vapour on nylon is estimated to be -18.0 kcal./mole. This is within experimental error both of the heat of absorption on cellulose acetate and of the heat of condensation to solid azobenzene.

Thanks are due to Lansil Ltd., British Nylon Spinners Ltd., and Imperial Chemical Industries Ltd. (Terylene Council) for gifts of cellulose acetate, nylon, and Terylene respectively. Thanks are due also to Dr. R. S. Bradley of the Department of Structural and Inorganic Chemistry, Leeds University, for helpful discussions on effusion measurements.

Much of the experimental work on which this paper is based was carried out by Mr. A. Hanrahan.

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The Dyeing of Acetate Rayon with Disperse Dyes V—Diffusion Coefficients in Cellulose Acetate Film

C. L. BIRD, F. MANCHESTER, and D. F. SCOTT

Values for diffusion coefficients, ranging from 0.05 to 4.8×10^{-16} cm.²/sec., have been determined at 80°c. for disperse dyes in cellulose acetate film. The results indicate that the rate of dyeing of disperse dyes is mainly governed by the rate of diffusion in the fibre.

INTRODUCTION

It is generally accepted that in a dyeing process at any particular temperature, with watersoluble dyes, the rate of dyeing is governed by the rate of diffusion in the fibre, provided that the movement of dye liquor is sufficient to maintain an equilibrium concentration of dye on the fibre surfaces and the conditions are such that a rapid "strike" is prevented. Thus Neale 1 found a good correlation when his values for the diffusion coefficients of a considerable number of purified direct dyes in cellulose film were compared with Boulton and Reading's 2 times of half-dyeing for direct dyes on viscose rayon. Boulton and Reading varied the amount of salt in their experiments so that the equilibrium exhaustion was only 50%, thus avoiding a rapid strike, which is caused with ionised dyes by the presence of an excess of added electrolyte. This danger does not arise with nonionic disperse dyes, provided that a stable

dispersion is used, but it is essential that the dispersion should be sufficiently fine for practically instantaneous dissolution of dye to take place 3. and so maintain a saturated solution, thus ensuring that diffusion is the rate-controlling step.

When a dyeing process is considered from the kinetic aspect, the fundamental properties of a particular dye in relation to the substrate are its diffusion coefficient and its activation energy of diffusion. This paper is mainly concerned with diffusion coefficients.

The following methods have been used by various workers to study the diffusion of dyes in fibres and films-

- Non-steady-state diffusion into both sides of a multiple membrane, using Fick's equation 5.
- Steady-state diffusion through a single membrane, using either (a) Fick's equation 5 or (b) Daynes' time-lag equation 6.

- (3) Non-steady-state diffusion into (a) pieces of film 7.8 or (b) fibres of circular crosssection 8-11, using the appropriate integrated form of Fick's equation.
- (4) Non-steady-state, edgewise diffusion into a film ¹², ¹³ or into a block of polymer ¹⁴, followed by measurement of the degree of penetration.

Methods (3) and (4) have been used with disperse dyes.

Vickerstaff ¹⁰ carried out dyeings at 80°c. with eleven commercial disperse dyes on nylon fibre, using an "infinite" dyebath. Diffusion coefficients were calculated by means of Hill's equation, and they varied from 0·1 to 2·3 × 10⁻¹⁹ cm.²/sec. Vickerstaff found no correlation between diffusion coefficient and rate of dyeing. From a series of dyeing experiments at different temperatures activation energies of 20–37 kcal./mole were obtained, the mean value being 23 kcal.

Kramer ¹⁴ used ten commercial (Perliton, BASF) disperse dyes, which were allowed to diffuse at 60°, 80°, and 100°c. into a block of Ultramid 6A (BASF, a copolymer of hexamethylene diadipate and e-aminocaprolactam). After dyeing, the block was cut into sections, and the degree of penetration z measured by means of a micrometer. The apparent diffusion coefficient D was obtained from the equation—

$$D = \frac{\bar{x}^2}{9a}$$

where x^2 is the mean square of the distance diffused, and t is the time. At 80° c. D varied between $8\cdot1$ and 36×10^{-8} cm.²/seè. The activation energy of diffusion varied between $16\cdot3$ and $22\cdot5$ kcal./mole, except for the most rapidly diffusing dye (Perliton Red 3B), for which it was $13\cdot6$ kcal.

With acetate rayon the serrated cross-section of the fibre prevents its use for diffusion experiments, but cellulose acetate films are readily prepared, so method 3(a) is suitable. This method has been employed by Majury \$, using five model substances, viz. p-nitroaniline, NN-dimethyl-p-nitroaniline azobenzene, p-aminoazobenzene, and NN-dimethyl-p-aminoazobenzene. Diffusion was carried out at 60° and 80°c. from a saturated solution, using both secondary acetate and triacetate film. The diffusion coefficients at 80°c. in secondary acetate varied from 2·9 to 200 × 10⁻¹⁰ cm.²/sec., and the activation energies of diffusion from 13 to 15 kcal./mole. With p-nitroaniline the diffusion coefficient was found to be independent of the concentration of the solution.

THE TIME-LAG METHOD

Instead of using method 3(a), it was decided to employ method (2), viz. steady-state diffusion through a cellulose acetate film, using for the interpretation of the results Daynes' time-lag equation ¹⁵—

$$D = \frac{d^3}{6L}$$

where D is the diffusion coefficient, d is the thickness of the film in cm., and L is the lag in sec. As

dye diffuses through the film it is removed at intervals and estimated colorimetrically. On plotting dye diffused against time a straight line is obtained, as illustrated in Fig. 1. The time lag L is the distance between the origin and the point where the straight line cuts the time axis. The method has the advantage over method 3(a) that rates of diffusion as well as diffusion coefficients can be measured, and the results are not rendered inaccurate by surface deposition of dye from unstable dispersions.

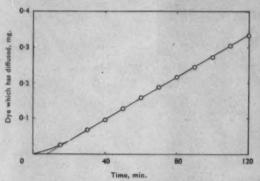


Fig. 1- Diffusion of 1:4-Diaminoanthraquinone

With the time-lag method it is essential that the concentration of the solution on the receiving side of the membrane should approach zero, but a measurable quantity of dye must be present. Many disperse dyes have an extremely low aqueous solubility 16, and diffusion through the film into water alone would quickly produce an almost saturated solution. At the same time the concentration of dye in the film at the exit side would approach saturation, instead of being very small. The experiments were therefore carried out with 0.002 m. sodium N-oleyl-4-anisidine-2-sulphonate on each side of the membrane. The micelles of the dispersing agent solubilise the dye as it diffuses out of the membrane, thus maintaining the desired concentration gradient in the film, as well as allowing measurable quantities of dye to accumu-

The weakness of the diffusion method chosen is that the results are very dependent on the thickness of the membrane, the time lag being proportional to the square of the thickness. In order to obtain a time lag of convenient magnitude with different dyes, the thickness of the membrane must be varied. It is seen from Table I that some of the rapidly diffusing dyes gave a rather short time lag, and greater accuracy would have been obtained with a thicker film.

With aminoazobenzene the rate of diffusion at 80°c. was so great that, with the thin films available, a concentration approaching zero could not be maintained on the receiving side of the membrane. The values obtained for the diffusion coefficient were obviously low, so Table I includes only a value for 60°c., which is in good agreement with that obtained by Majury *using method 3(a).

TABLE I

Diffusion Data for Disperse Dyes and Cellulose Acetate Film at 80°C.

Dye	Film Thickness (cm. × 10 ³)	Time Lag (min.)	(g./cm. ³ × 10 ⁶)	Rate of Diffusion* ds/ds g./sec. per cm. ³ × 10°)	Diffusion Co (cm. ² /sec. By Time-lag Equation	
4-Aminoarobenzene (at 60°C.)	2.08	48	17-6	23	5-2	4-0
4-Nitro-4'-aminoazobenzene	1.75 1.87	18 22	=	8·2 11·6	4·8 4·4	_
1-Amino-4-hydroxyanthraquinone	1-00	8	1-73	4-0	3-5	2.8
1-Methylaminoanthraquinone	1·82 1·62 1·09	32 23 26	-	2·2 2·5 2·6	2·9 3·2 · 3·0	=
1:4-Diaminoanthraquinone	0-83	8	1-68	3-3	2.4	1.6
1-β-Hydroxyethylaminoanthraquinone	1-45	25	3-24	5-5	2-4	2-5
2:4-Dinitro-4'-hydroxydiphenylamine	0-62 0-60	6-8 6-3	12·7 11·4	34 36	1·6 1·6	1·7 1·9
1:4-Diamino-2-methoxyanthraquinone	. 1·12 1·11	31 29	_	2·1 2·8	1·14 1·19	Ξ
4-Nitroaniline $\rightarrow NN$ -bis- β -hydroxyethylanilin	e 0-95 0-80	30 22	1.00 0.96	2·0 2·3	0.84	1.9
4-Nitroaniline $\rightarrow N$ -ethyl- N - β -hydroxyethylaniline	1.00 1.00 1.04	132 135 150	COURT COURT OF THE PARTY OF THE	1·03 0·92 0·70	0·76 0·74 0·70	-
4-Nitroaniline diethylaniline	0-90 0-90 1-10	36 36 51	0·43 0·45 0·48	0-36 0-36 0-28	0-62 0-62 0-66	0-75 0-72 0-64
1:4-Bismethylaminoanthraquinone	1·00 1·04	48 56	0·80 0·80	0·36 0·35	0·58 0·54	0·45 0·44
1-Methylamino-4- β -hydroxyethylaminoanthrouinone	n- 0-66. 0-64	45 49-5	_	1·1 · 1·6	0-27 0-23	-
2-Chloro-4-nitroaniline $\rightarrow N$ -ethyl- N - β -hydrox ethylaniline	y- 0.94 1.04	104 112	T	1·2 1·1	0·24 0·27	_
1:4-Bis(β -hydroxyethylamino)anthraquinone	0.87	100	6208	0-73	0-21	-
2-Methoxy-4-nitroaniline $\rightarrow NN$ -bis- β -hydroxy ethylaniline	7- 0-64 0-62	71 64	11-6 10-6	8·5 6·5	0·16 0·17	0·30 0·38
1-Methylamino-4-anilinoanthraquinone	. 1.00		-	0-02	ca. 0-05	_

* Obtained by dividing the slope (cf. Fig. 1) by the area in sq.cm. of the membrane

TABLE II

Diffusion Data for 1-Amino-4-hydroxyanthraquinone at Different Temperatures

Temp.		Film hickness m. × 10 ³)	Time Lag (min.)	Де (g./cm.³×1		Rate of Diffusion ds/ds /sec. per cm. × 10°)	Diffusion Co (cm. ² /sec. By Time-lag Equation	× 10 ¹⁰)
80		1.00	8	1.78		4-87	3-5	2-8
70		0-95	12	1.62		2-93	2.1	1.7
60		0-02	20	1-44		1.00	1.2	1-0
50		0.65	18	1.17		0-77	0.65	0.43
40		0.88	64	0-96		0.25	0-34	0.23
		Activa	ion energy o	of diffusion,	koal	./mole ·	. 12-5	14-9

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n s it Slowly diffusing dyes require the thinnest possible films, but such films are fragile, and with the very slowly diffusing 1-methylamino-4-anilino-anthraquinone it was impossible to use a sufficiently thin film.

The results of diffusion experiments with a series of purified disperse dyes are given in Tables I and II.

EXPERIMENTAL

Purified dyes and purified Lissapol LS (sodium N-oleyl-4-anisidine-2-sulphonate) were prepared as described in Part I ¹⁶. Solutions of purified Lissapol LS (ICI) were analysed by the method of Barr, Oliver, and Stubbings ¹⁷.

Cellulose acetate film was prepared by spreading an acetone solution of cellulose acetate on a glass plate and allowing the acetone to evaporate. The solution was prepared from a sample of cellulose acetate (Lansil) kindly supplied by Mr. H. A. Turner, and the procedure for preparing the film was very similar to that described by Daruwalla and Turner ¹⁸. After the film had been cut into discs of suitable size, it was washed to remove traces of acetone and stored under distilled water in a dark glass bottle.

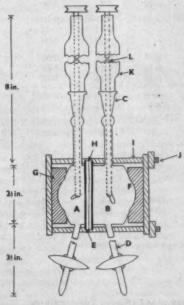


Fig. 2- Diffusion Cell

The diffusion cell, illustrated in Fig. 2, is a modified form of the cell described by Wright ¹⁹. The unit, constructed of Pyrex glass, consisted of two halves A and B, each of which was fitted with a standard joint C and a run-off tube D. At the junction of the two sections were ground glass flanges E. In order to achieve as small a capacity as possible and to allow the use of stirrers with maximum sweep, the end of each cell was extended in the form of a bulge F. This allowed the

stirrers to be placed centrally and so rotate close to the membrane. The bulges also provided a convenient anchorage for the rubber cushions G. The two halves of the cell were assembled with the membrane H between the two flanges, and the sections were held together by means of a brass frame I fitted with four clamping screws J. In order to obtain a watertight joint it was necessary to place the membrane between two polythene gaskets cut from 0.012-cm. sheet; and, to prevent the rubber cushions from sticking to the ends of the cell, two small pieces of cotton fabric were placed between the rubber and the glass. Each side of the cell was fitted with a small water-cooled condenser K, through which passed a link stirrer L. These stirrers, made from Pyrex rod and fitted with small stainless steel links, were driven independently at ca. 1000 r.p.m. by means of small electric motors.

The cell was contained in an air thermostat constructed from $\frac{1}{4}$ -in. asbestos sheet. The unit was heated by means of a number of woven asbestos resistance mats, and air circulation was achieved by means of a fan coupled directly to an electric motor. The inner temperature was controlled by means of a toluene–mercury regulator and a Sunvic hot-wire vacuum switch. Control between the limits of \pm 1°c. was obtained, which gave an internal temperature variation in the diffusion cell of \pm 0·25°c.

For the diffusion experiments, a piece of the cellulose acetate film of suitable size was mounted in the cell, after which the two compartments were filled with 0.002 m. sodium N-oleyl-4-anisidine-2sulphonate. In order to ensure that temperature conditions were stable, the cell was heated in the thermostat overnight. At the start of the run the solution at the low-concentration (left-hand) side of the cell was replaced by a fresh solution preheated to 85°c. Preheating was carried out in a wash bottle constructed from a Pyrex boiling tube, and the cell was quickly filled by blowing in the preheated solution. Trials showed that by the time filling was completed the temperature of the solution had dropped to 80°c. The requisite volume of dye dispersion (prepared in 0.002 m. sodium N-oleyl-4-anisidine-2-sulphonate as described previously 20) was also preheated to 85°c., and the experiment was started by running the solution from the right-hand side of the cell and refilling with the preheated dispersion. Filling was accomplished in less than 1 min., and timing was started when the cell was approximately half full.

When a measurable amount of dye had diffused, the left-hand compartment was emptied and refilled with preheated 0.002 m. sodium N-oleyl-4-anisidine-2-sulphonate. The solubilised dye was cooled to 20°c., made up to 50 ml. with acetone, and estimated in the usual way with a Spekker (Hilger) absorptiometer. By plotting the amount of dye diffused against time, curves of the type illustrated in Fig. 1 were obtained.

At the end of the diffusion run the liquor was removed from the cell and the dyed membrane was thoroughly washed in cold distilled water, dried between sheets of filter paper, and allowed to condition at 22°c. and 65% R.H. The average thickness of the membrane was then determined, as follows. About twelve circles (1.04 ± 0.01 cm. in diameter) were punched with a well sharpened corkborer. These were weighed on a semi-micro balance, and the thickness was calculated from the average weight of one circle, using a density figure of 1.33 g./c.c. No allowance was made for the swelling of cellulose acetate film in water, but Marsden and Urquhart's data at indicate that, at 80°c., the absorbed water is nearly all located in pores and that the degree of swelling will be very small. The effective area of the membrane was calculated from the diameter of the circular hole in the polythene gasket.

In an additional experiment a film was used which had previously been dyed by allowing 1-methylamino-4-anilinoanthraquinone to diffuse through it for 24 hr. After the dispersion of this dye had been removed, and the cell rinsed thoroughly, a diffusion experiment was carried out in the usual way with a dispersion of 1-amino 4-hydroxyanthraquinone.

Instead of using Daynes' time-lag equation for calculating the diffusion coefficient, it is possible to use Fick's equation—

$$\frac{\mathrm{d}s}{\mathrm{d}t} = -D \frac{\Delta e}{d}$$

where ds/dt is the rate of diffusion in g./sec. per cm.², D the apparent diffusion coefficient in cm.²/sec., d the thickness of the membrane in cm., and dc the difference in concentration of dye across the membrane in g./cm.³. If the concentration of dye in the film is determined after the diffusion experiment, the value obtained will correspond to $\frac{1}{2}dc$, assuming that the concentration across the thickness of the film changes in a uniform manner

from saturation to zero. Some of the dyed films which had been retained after dissection were analysed, and the diffusion coefficients obtained by this method are given in Table I. It is seen that, although the results are of similar magnitude and in nearly the same order as that shown by the time-lag method, the overall range has contracted.

DISCUSSION

The factors most likely to affect the rate of diffusion of a disperse dye through cellulose acetate are—

- (1) The size of the molecule
- (2) The affinity of the dye
- (3) The saturation value.

With acetate rayon the molecular size is quite critical 23, large dye molecules being unable to penetrate the fibre owing to its low degree of swelling. The low diffusion coefficient of 1-methylamino-4-anilinoanthraquinone may well be largely due to the bulky phenyl group attached to the 4-position. In this connection it may be noted that Vickerstaff 23 found 1-ethyl- and $1-\beta$ -hydroxyethyl-amino-4-anilinoanthraquinone to be as slowdyeing as the 1-methyl derivative, whilst Kartaschoff and Farine 24 found that 1-amino-4p-toluidino- and 1:4-di-p-toluidino-anthraquinone would scarcely penetrate acetate rayon at 60°c. To a lesser extent the same effect is seen when the diffusion coefficient of 1:4-diaminoanthraquinone is compared with those of XII, XV, and XVI in Table III. Some indication of molecular size is given by the molecular weight, when compounds of similar structure are compared, and, as shown in Table III, there is a correlation between the molecular weights of disperse dyes and their diffusion coefficients in cellulose acetate film. A similar correlation was observed by Vickerstaff 10 for disperse dyes and nylon fibre.

TABLE III

Relation between Diffusion Coefficient and Molecular Weight

No.	Dye	Mol. Wt.	$0 \text{ (cm.}^{5}/\text{sec.} \times 10^{10})$	Partition Coefficient at 80°c.
	Azo (and Nitrodiarylamine)			
I	4-Aminoazobenzene	. 197	10-6	500 4
п	. 4-Nitro-4'-aminoazobenzene	. 242	4.6	1,400
III	2:4-Dinitro-4'-hydroxydiphenylamine	. 275	1.6	510
IV	4-Nitroaniline→diethylaniline	298	0.63	12,000
V	4-Nitroaniline $\rightarrow N$ -ethyl- N - β -hydroxyethylaniline	. 314	0.73	2,300
VI	4-Nitroaniline $\rightarrow NN$ -bis- β -hydroxyethylaniline	. 330	0.82	530
VII	2-Chloro-4-nitroaniline -> N-ethyl-N-β-hydroxyethylaniline	348-5	0.26	5,500
VIII	2-Methoxy-4-nitroaniline $\rightarrow NN$ -bis- β -hydroxyethylaniline	360	0.17	430
	Anthraquinonoid			
IX	1-Methylaminoanthraquinone	. 237	3-0	1.400
X	1:4-Diaminoanthraquinone	. 238	2-4	560
XI	1-Amino-4-hydroxyanthraquinone	. 239	3.5	910
ХП	1:4-Bismethylaminoanthraquinone	. 266	0.56	2,300
XIII	1-β-Hydroxyethylaminoanthraquinone	. 267	2-4	500
XIV	1:4-Diamino-2-methoxyanthraquinone	. 268	1-17	1,100
XV	1-Methylamino-4-β-hydroxyethylaminoanthraquinone	. 296	0.26	730
XVI	1:4-Bis(β-hydroxyethylamino)anthraquinone	. 326	0.21	180
XVII	1-Methylamino-4-anilinoanthraquinone	328	ca. 0-05	Very high

The diffusion coefficient of 1-amino-4-hydroxy-anthraquinone diffusing through a film dyed with 1-methylamino-4-anilinoanthraquinone was, within the limits of experimental error, the same as that obtained with undyed film. It thus appears that these two dyes diffuse independently, and do not form a molecular compound, as is the case with some pairs of disperse dyes 3.25.

Contrary to Majury ⁸, we find little sign of any correlation between diffusion coefficient and affinity, as shown in Table III. The partition coefficients K given in this table, which are a measure of affinity, were determined by Miss Pamela Harris ²⁶; they refer to partition between accetate rayon and water, i.e. no dispersing agent was present. The partition coefficient is defined by—

$$K = \frac{D_{f}}{D_{s}}$$

where $D_{\rm t}$ is the concentration of dye in the fibre and $D_{\rm s}$ its concentration in the aqueous solution, expressed in moles per kilogram of completely dry fibre and moles per litre, respectively. Comparing IV and V, which have almost the same molecular size, it is seen that, although the diffusion coefficients are similar, IV has a much greater affinity than V. Similarly with XII, XV, and XVI; XII has the highest affinity, yet it has the highest diffusion coefficient.

It may be significant that the dyes IV and XII, which have comparatively high diffusion coefficients, are relatively volatile, and they may not be very firmly attached to the fibre. Their apparent high affinity is due not so much to high affinity for cellulose acetate as to low affinity for water (low aqueous solubility ¹⁶). If diffusion of disperse dyes can take place by dye molecules moving from site to site, e.g. along the walls of the

pores, without first dissolving in water, the difficulty is overcome. With dyes which are fairly soluble and are not volatile, e.g. V, the normal process of diffusion through the internal aqueous phase will predominate. Evidence in support of this view is provided by the ability of some disperse dyes to dye acetate rayon from the vapour phase, and by the suitability of the more volatile disperse dyes for colouring the hydrophobic fibre Terylene, where the conditions are unfavourable for diffusion of dye molecules through water-filled pores. Some indication that affinity plays a part is given by V-VII, but with VIII it would appear that the 2-methoxy group hinders diffusion, since this dye has low affinity.

It is not possible to compare diffusion coefficients with rates of dyeing unless an approximately constant concentration gradient is maintained. In Boulton and Reading's 2 time-of-half-dyeing experiments this was done by limiting the equilibrium exhaustion to 50%. With disperse dyes it is necessary to compare rates of diffusion (not diffusion coefficients) with rates of dyeing. The relationship between rate of diffusion and diffusion coefficient D is given by the equation—

$$P = D \times \text{Solubility}$$

where P, the permeability, is the rate of diffusion for a film of 1 cm. thickness, i.e.—

$$P = \frac{ds}{dt} \times d$$

The solubility, or saturation value, is the amount of dye adsorbed by cellulose acetate when it is in equilibrium, at a particular temperature, with a saturated aqueous solution of the dye. In Table IV rates of diffusion (permeabilities) are compared with commercial ratings for speed of dyeing and with Vickerstaff's times of half-dyeing ²³. The results show a fairly good correlation, and it is

TABLE IV

Comparison of Rates of Diffusion with Rates of Dyeing

Dye	Rate of Diffusion at 80°c. ds/dt \times d (g./sec. per cm. \times 10 ¹¹)	Rate of Dyeing of Commercial Dye*	Time of Half-dyeing at 85°C. ²¹ (min.)
2:4-Dinitro-4'-hydroxydiphenylamine	2.14	R	0.75
4-Nitro-4'-aminoazobenzene	1.81	R	0.8
1-β-Hydroxyethylaminoanthraquinone	0.79	M-R	
1-Amino-4-hydroxyanthraquinone	0.49	R	2.4
1-Methylaminoanthraquinone	0.42	M	_
2-Methoxy-4-nitroaniline -> NN-bis-β-hydroxyethylaniline.	. 0.38	M-R	-
1:4-Diaminoanthraquinone	0-27	R	3.0
1:4-Diamino-2-methoxyanthraquinone	0-27	R	4-8
4-Nitroaniline $\rightarrow NN$ -bis- β -hydroxyethylaniline	0.19	M	3.3
4-Nitroaniline -> N-ethyl-N-β-hydroxyethylaniline	0.18	M	4.0
2-Chloro-4-nitroaniline $\rightarrow N$ -ethyl- N - β -hydroxyethylaniline	0-11	8	16-5
1-Methylamino-4-β-hydroxyethylaminoanthraquinone .	0.09	M	
1:4-Bis(β-hydroxyethylamino)anthraquinone	0.064	8	-
1:4-Bismethylaminoanthraquinone	0-036	S-M	16-5
1-Methylamino-4-anilinoanthraquinone	0.002	8	30

concluded that rate of dyeing is largely determined by rate of diffusion in the fibre. With some disperse dyes, however, e.g. with Duranol Blue 2G, the rate of dissolution of the crystalline dye particles may be the controlling factor, especially

when dyeing at low temperatures 20.

The distinction between diffusion coefficient and rate of diffusion is illustrated by 1:4-bismethylaminoanthraquinone. This is a slow-dyeing disperse dye, the relatively high diffusion coefficient being counteracted by the low saturation value, which results in a low concentration gradient. On the other hand, with 2-methoxy-4-nitroaniline-> NN-bis-β-hydroxyethylaniline, a low diffusion coefficient is counterbalanced by a high saturation value.

With 1-amino-4-hydroxyanthraquinone diffusion experiments were carried out over a range of temperatures, and the activation energy of diffusion was calculated in the usual way from the slope of the straight line obtained by plotting the logarithm of the diffusion coefficient against the reciprocal of the absolute temperature. The value obtained (Table II) was 12.5 kcal./mole, which is not far removed from Majury's values (13-15 kcal.).

On the basis of the previous arguments concerning diffusion coefficients and rates of diffusion, it would seem that activation energies of dyeing, as determined by Vickerstaff 10, should be compared, not with activation energies of diffusion, but with "activation energies of rate of diffusion", obtained by plotting log $(ds/dt \times d)$ against 1/T. The activation energy of the rate of diffusion of 1-amino-4hydroxyanthraquinone was found to be 18-2 kcal./mole, which is not far removed from the average value of Vickerstaff's activation energies of dyeing for disperse dyes on nylon (23 kcal.).

With direct dyes on cellulose the diffusion coefficient is not constant, but varies with the concentration of the dye solution 5. On the other hand, Majury 8 has shown that the diffusion coefficient of p-nitroaniline in cellulose acetate is independent of concentration. We have not investigated this problem thoroughly, but the result of a single experiment (in triplicate) confirmed Majury's finding. Instead of using a dispersion of p-nitroaniline $\rightarrow N$ -ethyl-N- β -hydroxyethylaniline, a fairly dilute (20 mg./litre) solubilisate in 0.002 m. sodium N-oleyl-4-anisidine-2-sulphonate was used, the solubilisate being changed at frequent intervals so as to maintain a constant concentration at the membrane surface. The value obtained for the diffusion coefficient was $0.49~\times~10^{-10}$ cm. $^2/\mathrm{sec.}$, which is lower than the value in Table I, but almost identical with that obtained with a dispersion which, like the solubilisate, had been preheated for 24 hr. before use. The lower values are probably connected with the slight decomposition which occurs on prolonged heating of a solution of sodium N-oleyl-4-anisidine-2-sulphonate 27.

Standing, Warwicker, and Willis 28 have shown that, with cellulose and direct dyes, the apparent diffusion coefficient is proportional to the slope of the adsorption isotherm. With disperse dyes this slope is constant, so the diffusion coefficient should be independent of concentration.

In the early stages of our work on disperse dyes it was thought that dispersing agents would be unable to penetrate acetate rayon because of their large molecular size. By carrying out a diffusion experiment for nine days, however, it was found that diffusion through the film did take place with Lissapol LS. Owing to the difficulty of measuring very small amounts of dispersing agent, compared with the ease with which dyes can be measured, it is not possible to give a numerical value for the diffusion coefficient, but it can be stated that Lissapol LS (sodium N-olevl-4-anisidine-2-sulphonate) behaves like a very slow-dyeing disperse dve. It is unlikely that the very small amount of dispersing agent present will have any appreciable influence on the diffusion of dyes inside the fibre.

Thé authors are indebted to Dr. A. B. D. Cassie. Director of Research of the Wool Industries Research Association, for allowing Mr. F. Bond to construct the diffusion cell in the laboratories of the Association. They also thank Dr. M. L. Wright, formerly of the W.I.R.A., for much valuable advice. The dyes used in this work were kindly supplied by British Celanese Ltd., the Dyestuffs Division of Imperial Chemical Industries Ltd., and the Yorkshire Dyeware & Chemical Co. Ltd.

DEPARTMENT OF COLOUR CHEMISTRY AND DYEING

THE UNIVERSITY LEEDS 2

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A Note on the Diffusion of Disperse Dyes in Nylon Yarn

C. L. BIRD and D. F. SCOTT

Diffusion coefficients and times of half-dyeing have been determined for three commercial anthraquinonoid disperse dyes on nylon yarn. The results support the view that rate of dyeing is mainly governed by rate of diffusion of dye in the fibre.

INTRODUCTION

When the results of Bird, Manchester, and Scott¹ for the diffusion coefficients of disperse dyes in cellulose acetate film are compared with those of Vickerstaff² for disperse dyes in nylon yarn, it is seen that the two sets of data show fairly good agreement with six of the eight dyes for which a comparison can be made. Of the remaining two dyes, Duranol Red G (1-methylaminoanthraquinone) appears to diffuse much more slowly in nylon than in cellulose acetate, whilst Duranol Blue 2G was found by Vickerstaff to be the most rapidly diffusing of the six anthraquinonoid dyes which he examined.

Since Duranol Blue 2G diffuses extremely slowly in cellulose acetate and dyes both acetate rayon and nylon very slowly, it seemed to us unlikely that Vickerstaff's figure for the diffusion coefficient in nylon could be correct. We therefore repeated his diffusion and rate-of-dyeing experiments with the following three commercial anthraquinonoid disperse dyes—

Duranol Violet 2R (1:4-diamino-anthraquinone)

Duranol Red X3B (1:4-diamino-2-methoxyanthra-quinone)

Duranol Blue 2G

Our results, together with those of Vickerstaff, are given in Tables I and II.

NYLON—The yarn used, kindly supplied by British Nylon Spinners Ltd., was bright 210/34. It was extracted in a Soxhlet for 24 hr. with petroleum ether (40-60°c.), dried, washed in water, dried, and conditioned at 65% R.H. and 22°c.

DYES—Three commercial dyes, kindly supplied by I.C.I. Ltd. Dyestuffs Division, were used, viz. Duranol Violet 2R 300, Duranol Red X3B 300, and Duranol Blue 2G 300. The respective strengths of these dyes, based on colorimetric comparison against purified samples, were found to be 32%, 16.5%, and 11.5%.

RATE OF DYEING—Dyeings were carried out at 80°c. in a modified Marney machine 3 for varying times, using 1.5-g. hanks of purified and conditioned yarn and a liquor: yarn ratio of 50: 1. The dyebath contained 0.2 g. of commercial Lissapol LS (ICI) per litre. With Duranol Violet 2R a 1.5% dyeing was used; the percentages of the other two dyes were adjusted so as to give approximately the same amount of pure dye. For times up to 10 min. dyeings were done in triplicate and for longer times in duplicate.

DIFFUSION—A dyebath containing 0.5 g. dye and 2.0 g. purified Lissapol LS per litre was used, with 0.1 g. hanks of purified and conditioned yarn and a liquor: yarn ratio of 800: 1. Dyeings in triplicate were carried out for 10 min., 20 min., and

TABLE I

Dye		Diffusion Coefficient (cm. 2/sec. × 1010)						Saturation Value (%) (from 24-hr. dveing)			
		From 10-min. Dyeing	rom From 20-min.		Yarn Mean	Vickerstaff ²	Cellulose Acetate Film	Found Vickerstaff ²			
Duranol Violet 2R 300		2.8		2-4	2.6	1.1	2-4	3.9	4.9		
Duranol Red X3B 300		1.8		1.8	1-8	1.5	1.2	4-9	7-6		
Duranol Blue 2G 300		0.55		0-49	0.52	1.7	en. 0-05	3-4	3-6		

	TABLE II		
	Dye	(1	f Half-dyein min.) Vickerstaff
1.5%	Duranol Violet 2R 300	3.3	4-5
2.9%	Duranol Red X3B 300	6.8	-
3.5%	Duranol Red X3B 300	-	15
4.2%	Duranol Blue 2G 300	75	
3.0%	Duranol Blue 2G 300	-	60

EXPERIMENTAL

The experimental conditions were not quite the same as those in Vickerstaff's experiments. In particular, the denier of the yarn was different, the filament radius being 14×10^{-4} cm., compared with 9×10^{-4} cm. in Vickerstaff's experiments.

24 hr., and the diffusion coefficient was calculated in the usual way with the aid of Hill's equation.

Colorimetric Estimation—The dyed yarn, after rinsing, drying, conditioning, and weighing, was dissolved in freshly distilled o-chlorophenol, and the concentration of dye determined by means of a Spekker (Hilger) absorptiometer, a blank containing an equivalent amount of undyed yarn being used.

DISCUSSION

In a preliminary series of diffusion experiments dispersing agent was omitted from the dyebath, as in Vickerstaff's experiments, but after dyeing for 24 hr. with Duranol Blue 2G the dispersion had coagulated. This would clearly result in

NOTES

precipitation of dye on the surface of the fibres and a high saturation value. A series of 24-hr. dyeings was therefore carried out, as in the diffusion experiments, but with increasing amounts of Lissapol LS. It was found that an addition of 2 g. of purified Lissapol LS per litre stabilised the Duranol Blue 2G suspension, so the diffusion experiments were repeated with all three dyes using this addition. Under these conditions less dye was taken up at all three times, particularly with Duranol Blue 2G, but the alteration in the diffusion coefficient was comparatively small. Judging from the saturation values (Table I), no coagulation of the dispersion occurred with Duranol Blue 2G in Vickerstaff's experiments, but the higher values for the Violet 2R and Red X3B indicate incompletely stabilised suspensions.

Compared with Vickerstaff's results, our results differ mainly in respect of the diffusion coefficient of Duranol Blue 2G; our value is in line with the

diffusion coefficient in cellulose acetate and with the rates of dyeing on cellulose acetate and nylon. We also find the diffusion coefficient of Duranol Violet 2R to be greater than that of Duranol Red X3B. It seems probable that, as with cellulose acetate ¹, the factor governing the rate of dyeing of disperse dyes on nylon is, in general, the rate of diffusion of dye in the fibre.

DEPARTMENT OF COLOUR CHEMISTRY AND DYEING THE UNIVERSITY LEEDS 2

(Received 18th October 1955)

References

- Bird, Manchester, and Scott, J.S.D.C., 72, 49 (Feb. 1956).
- Vickerstaff, ibid., 59, 92 (1943); The Physical Chemistry of Dyeing (London: Oliver & Boyd. 2nd edition 1954), pp. 442 and 449.
- 8 Bird, J.S.D.C., 70, 68 (1954).

Notes

Meetings of Council and Committees January

Council-11th

Publications-17th

Colour Index Editorial Panel-4th and 18th

Perkin Main-16th

Terms and Definitions-19th

Death

We regret to report the loss by death of M. Louis Discrens.

A.S.D.C. Examination

It is intended to hold an examination for the Associateship of the Society of Dyers and Colourists on Thursday-Saturday, 14-16th June 1956. Completed application forms (obtainable from the Society at 19 Piccadilly, Bradford 1, Yorkshire) must reach the Society not later than 31st March 1956.

Report of the Society's Medals Committee

At a meeting of Council held at the Victoria Hotel, Bradford, on 11th January 1956, the following recommendations of the Society's Medals Committee were unanimously approved.

Firstly, the Committee recommended that-

The Perkin Medal be awarded posthumously to Dr. W. H. Carothers for the discovery of nylon.

The Perkin Medal be awarded to John R. Whinfield, C.B.E., M.A., F.R.I.C., for the discovery of Terylene

The Committee also recommended that, the work of the Fastness Tests Committees having approached finality—

A Bar to the Gold Medal be awarded to P. W. Cunliffe, Ph.D., F.R.I.C., F.T.I., F.S.D.C., for continued valuable services to the Society as Chairman of the Fastness Tests Co-ordinating Committee

The Silver Medal be awarded to K. McLaren, B.Sc., F.R.I.C., F.S.D.C., for valuable services to the Society as Honorary Secretary of the Fastness Tests Co-ordinating Committee

The Silver Medal be awarded to J. G. Grundy, F.S.D.C., for valuable services to the Society as Chairman of the Washing Fastness Subcommittee

The Silver Medal be awarded to M. E. Probert, Ph.D., F.R.I.C., for valuable services to the Society as Chairman of the Bleaching Fastness Subcommittee

The Silver Medal be awarded to E. Wilson, B.A., F.S.D.C., for valuable services to the Society as Chairman of the Alkaline Milling, Burnt Gas Fumes, and Perspiration Fastness Subcommittees.

The Medals Committee further recommended that a Diploma expressing the thanks of the Council for services rendered be awarded to the other members of the various Fastness Tests Co-ordinating Committee and the various Fastness Subcommittees.

It was also recommended that-

The Silver Medal be awarded to H. W. Ellis, Ph.D., A.R.C.S., A.R.I.C., F.S.D.C., for valuable services to the Society as Honorary Secretary of the London Section from 1938 to 1954

The Silver Medal be awarded to J. V. Summersgill, B.Sc., F.S.D.C., for valuable services to the Society as Honorary Secretary of the Publications Committee from 1945 to date.

Bicentenary of the Birth of Jean Antoine Claude Chaptal

Chaptal (1756-1832) was the last of the great French dye chemists of the eighteenth century. Having studied in Paris, he was appointed at the age of 25 professor of chemistry at Montpellier, but his interests were almost exclusively in applied and not theoretical chemistry. He established a factory at Montpellier which met all the French dyers' needs for alum, and he later obtained interests in chemical and textile factories in other parts of France. When Chaptal started work, cotton dyeing was regarded as a most difficult process, but his investigations led to considerable simplification in dyeing methods. His book L'Art de la Teinture du Coton en Rouge (Paris 1805) was the first to pay due regard to the constitution of cotton as a vegetable fibre. He became Minister of the Interior under Napoleon, organised the first official industrial exhibitions in Paris in 1798 and 1801, and founded the École (now the Musée) des Arts et Métiers. He was the foremost practical chemist in France of his day, occupying the same place in applied chemistry as Berthollet occupied in theoretical chemistry. C. O. C.

Charles Frederick Cross, F.R.S., 1855-1935

The centenary of the birth at Brentford, Middlesex, of C. F. Cross, co-inventor of the viscose process, occurred on 11th December 1955. After graduating at King's College, London, he studied at Zürich Polytechnic and then at Owens College, Manchester, where he met Edward J. Bevan. Viscose was discovered by the two chemists in their laboratory at Kew, and was patented in 1892. Cross was also a pioneer of the applied chemistry of cellulose, of viscose films, and of cellulose acetate. He received the Society's Perkin medal in 1923, became an Honorary Member in the same year, and was President during 1918–1920.

New Books and Publications

Physics of Fibres An Introductory Survey

By H. J. Woods. Pp. 100. London: Institute of Physics. 1955. Price, 30s. 0d.

This is the most recent in the series of specially commissioned monographs on particular branches of applied physics published under the general title of Physics in Industry. The author has aimed at providing an introduction to the subject for scientists commencing research and development work in this field; he has also had in mind the scientists whose interests are in other fields, but who may wish to learn something about the ways in which physics has helped in advancing knowledge of fibres. The six chapters deal with fibre structure in general, general physical properties, X-ray diffraction, optical and elastic properties, and electron microscopy; a bibliography and a fairly full list of references are provided. The subject matter is chiefly concerned with physical techniques, and the emphasis throughout is on the natural rather than the man-made fibres.

This is a scholarly book, lucidly written by an author with intimate knowledge of the subject. Scientists whose interests are in other fields would probably have appreciated more illustrations well known to textile scientists, for example of fibre cross-sections and "absorption isotherms", but this criticism is mitigated by the descriptive text. As an introductory survey of a specialised field the book is excellent.

W. Guy

Fibre to Fabric

By M. D. Potter and B. P. Corbman. 2nd edition 1954. Pp. viii + 344. New York: McGraw-Hill Book Co. Inc. Price, 23s. 0d.

The purpose of this book is stated in the preface to be to meet the vocational needs of students expecting to enter the distributive trades and to provide a basic complete textile book. The first of these it may possibly achieve to a certain extent, but the second it can scarcely be said to do. The order of presentation follows the usual method of considering fibre structure and chemical and physical properties before dealing with processing, and this section may be regarded as of some value to the retailer; but, since it is entirely free from serious scientific study, its value in the modern school of textile teaching is very limited.

The sections on spinning, weaving, and finishing are, again, too limited in scope and too vague in detail to be of value to the serious student and, indeed, too inaccurate in some cases to be of assistance to any student. Dyeing is dismissed in some thirteen pages containing such loose statements as "basic dyes are direct dyes" and "azo dyes... are used as acid dyes".

The section on man-made fibres is fairly full and up to date and would be useful for reference, as would the glossary of viscose rayon and acetate rayon fabrics. It must be borne in mind that the book is primarily intended for use in the U.S.A., and allowance must be made for variations in the meaning of terms. The effect is to create in the mind of English readers a certain amount of confusion and a sense of vagueness, which is not lessened by a tendency to sweeping statements about fibre properties and processing. In a second edition most of these inaccuracies should have been corrected.

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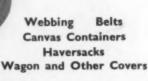
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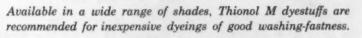
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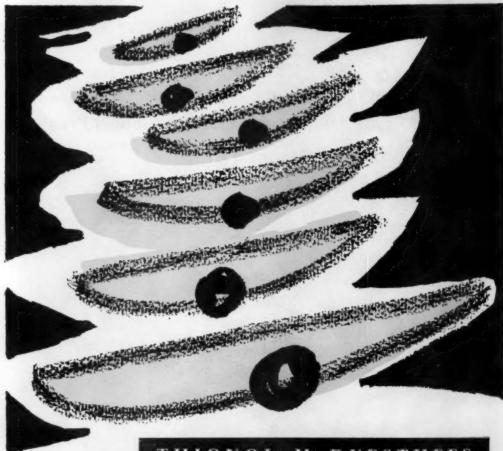
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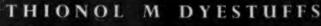
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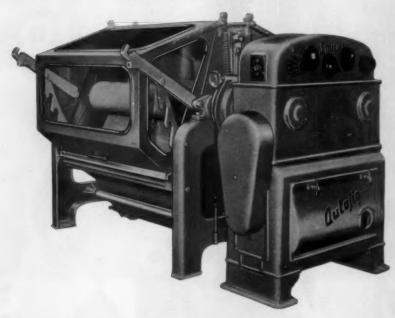
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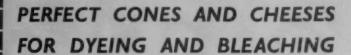
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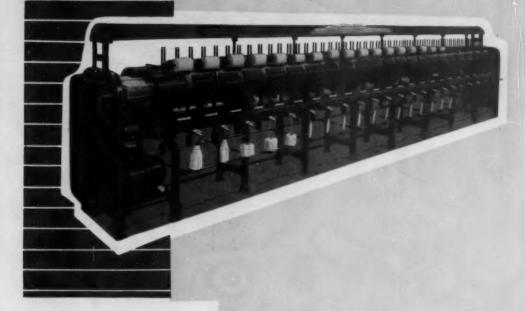
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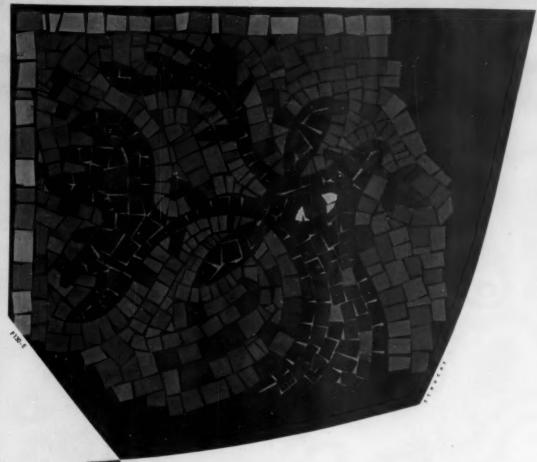
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Abstracts from British and Foreign Journals and Patents

The titles of abstracts may be modified. Abbreviations of names of firms are listed in J.S.D.C., 68, 23 (Jan. 1952), and also, together with symbols and the periodicals abstracted, in the annual index

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C.O.C.

Alignment Control Device for Moving Webs atronzellstoff- und Papierfabriken AG. BP 741,320

Weft-straightening Mechanism for Tenters Macknight & Hoffman USP 2,701,405

Hygrometer Arrangement for Measuring the Humidity of Moving Webs

Paul Lippke BP 741.164 Maintaining Constant Tension in Fabrics during

James Hunter Machine Co. USP 2,701,717

The fabric is engaged by a resilient roller having a yieldable periphery. This roll is driven at a constant speed, but as it is deformed by the tension in the fabric its peripheral speed varies inversely with the fabric tension. This roll coacts with means for feeding the fabric to it at a linear speed less than the peripheral speed of the roll at its maximum radius but greater than that of the roll at its minimum radius. Thus the tension in the fabric is maintained constant by the radius of the roll varying in accordance with the tendency of the fabric tension to increase or diminish.

Fibre Bowls which do not readily Dry Out

Richard Hough BP 740,769 A calender or mangle bowl consists of compressed wood fibres rigidly mounted co-axially upon a shaft. There are passages in the shaft through which liquid may be fed to impregnate the surrounding mass of wood fibres. Thus the · moisture lost by the fibres can be constantly replenished; this results in the bowl having a much longer life.

Tension Control of Sheet Material being Unwound

Beloit Iron Works A Vertical-roll Impregnating and Coating Machine General Motors Coron. USP 2,700,620 General Motors Corpn.

The cloth or paper to be impregnated is guided in a vertical position between two vertical applicator rolls, which move the cloth or paper and evenly impregnate or coat both sides of it.

Coating Sheet Material with Powder, Granules, etc.

The powder or the like is dropped vertically towards the sheet (which has been previously coated with an adhesive) and is then forcibly diverted in a horizontal direction so that it falls on the sheet in uniform concentra-C.O.C.

Cloth-folding Machine United Merchants & Manufacturers USP 2.701.715

Agitator for Cloth-washing and Homogenising Machines

Kovotechna An impeller having a worm-like body having one or more flights but no core rotates within the washing or mixing chamber so that the liquid moves from the base to the top of the impeller. As a substantial proportion of the liquid is expelled sideways from the impeller, clothes are prevented from coming into close contact with the flights of the worm. C.O.C.

Rack for Dyeing Stockings A. E. Williams and J. A. Leger USP 2,701,460 A wheel carrying the racks on its circumference rotates vertically in a dye tank so that the racks pass into and out of the liquor repeatedly in succession. The stockings are held in the racks by plates which block off part of the fabric from the dye liquor. By changing the dye and at the same time changing the plates for those of another pattern, multicoloured patterns are produced on the stockings.

Garment-pressing Machine

Pantex Manufacturing Co. BP 741,340

Electrostatic Spray-coating Apparatus Harper J. Ransburg Co.

BP 741,313 Coating Webs of Non-absorbent Material with Liquid

simultaneously on Both Sides L. van der Meulen

Metering Pumps for Use in Colour Printing, etc. McCorquodale Colour Display BP 741,902

Transfer Gripper System for Rotary Multicolour Printing Machines for Sheets of Tin Plate or the like

A system which prevents a sheet which is not held, or is not held at the correct time, disturbing the operation of or damaging the machine. C.O.C.

Continuously Milling Plastics Union Carbide & Carbon Corpn.

BP 741,532

Transfers and Apparatus for making and applying them (IX p. 72)

Spraying Loose Wool with Sighting Colours and/or Oil (X p. 73)

II— WATER AND EFFLUENTS

Treatment of Dyeworks Effluents-II R. Baetsle, E. Leclerc, L. Ghyssaert, and J. Verbeke Bull. centre belge étude et document (Liège)

No. 27, 16-29 (1955)

Chem. Abs., 49, 14327 (25 Oct. 1955) Effluent from a medium-sized dyeworks should be treated with FeCl_a (300 mg./l.) to give a ppt. which decants easily. As this method gives large quantities of sludge, large dyeworks should use bacteria beds. Thus sludge, large dyeworks should use bacteria beds. Thus 9 cu.m. of water can be pumped through 1 cu.m. of bacteria bed/day. B.O.D. is decreased by approx. 75%, e.g. 5,500 cu.m. of effluent/day need a bacteria bed 20 m. in diameter and 2 m. deep. The amount of free Cl. which varied from 0.05-0.75 mg./l. was reduced to enable acclimatisation of the bacteria. Although first cost is high, the working costs are low.

III-CHEMICALS: AUXILIARY PRODUCTS; FINISHING MATERIALS

Benzylation of Sulphanilic Acid

V. F. Borodkin

J. Appl. Chem. U.S.S.R., 28, 777-780 (July 1955) A study is made of the conditions necessary for the benzylation of sulphanilic acid with the formation of products (Solution Salts) containing a predominance of the mono- or di-benzyl deriv., as desired, together with as little sodium sulphanilate as possible.

Modification of the Drying Properties of Oils. III- Halogenation

XXVIII Congrès International de Chimie Industrielle Chim. et Ind., 74, 173 (Oct. 1955)

Introduction of chlorine groups into acyl radicals followed by elimination of hydrochloric acid creates new double bonds, resulting in a marked increase in the conjugation and the drying properties of oils. S.R.C.

Stabilisation of Polyethylene Glycol Ethers

Chakhovskoy and P. Lambert

XXVIII Congrès International de Chimie Industrielle

Chim. et Ind., 74, 184 (Oct. 1955) Hexaethylene glycol stearates prepared in presence of NaOH behave undesirably in hot acid aqueous emulsion because of the presence of soaps, and they show appreciable volatility at 100°c. Both defects are diminished by treatment with PCl₃ followed by neutralisation, by addition of hexaethylene glycol distearate, or by addition S.R.C. of hexaethylene glycol stearate-phosphate. .

Indigenous Tanning Materials of Madras State.

I— White Valem Bark
J. B. Rao, Y. Nayudamma, and B. M. Das

J. Sci. Ind. Research (India), 13 B, 867-873 (1954)

PATENT

Surface-active Compositions containing 4-Methyl-7-diethylaminocoumarin as a Fluorescent Brightening

BP 741,959 Addition of \$\leq 5\%\$ by weight of 4-methyl-7-diethyl-aminocoumarin to a surface-active agent yields products which are readily soluble in water. C.O.C.

Dewatering of Aqueous Pastes or Slurries of Finely Divided Hydrophobic Solids (IV p. 67)

IV-RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS

Common Basis of Organic Oxidations in Acidic Solution

L. S. Levitt

J. Org. Chem., 20, 1297-1310 (Oct. 1955)
(a) The positively charged atom of the oxidant always attacks a free electron pair of the reductant at a centre of high electron density, forming a coordinate bond, and the atom initially attacked, occasionally the adjacent one, becomes positively charged.

(b) The positive charge is removed by expulsion of a positive fragment, usually a proton, or by combination

with an available anion.

The oxidiser begins to move off with the electron pair of the coordinate bond, resulting in a transition state in which the newly forming positive organic species is simultaneously expelling a second positive fragment or combining with a second negative ion. 70 references.

Conversion of Nitriles to Amides by means of Boron Fluoride

C. R. Hauser and D. S. Hoffenberg J. Org. Chem., 20, 1448–1453 (Oct. 1955)

Chlorination of Acetanilide

M. Pinar Martinez

XXVIII Congrès International de Chimie Industrielle Chim. et Ind., 74, 180 (Oct. 1955)

The reaction was studied using chlorine in aqueous solution at various concentrations, giving a 70% yield of p-chloroacetanilide with chlorine conen. of 3%. Chlorination in (a) aqueous suspension, (b) sulphuric acid, (c) benzene, chlorobenzene, nitrobenzene, carbon tetrachloride, and chloroform gives limiting yields of (a) 30%. S.R.C. (b) 50%, (c) 60%.

Preparation 2:4-Dichlorophenol Applications H. Ferrer Andreu

XXVIII Congrès International de Chimie Industrielle Chim. et Ind., 74, 178 (Oct. 1955)

Various methods of preparation from readily available sterials have been studied. S.R.C. materials have been studied.

Nature of the Internal Hydrogen Bond A. Burawoy, M. Cais, J. T. Chamberlain, F. Liversedge, and A. R. Thompson

II—Electronic Spectra of 2-Nitrosoanisole and 6-Methoxyiminocyclohexa-2:4-dienone (o-Benzo-quinone 1-Monoxime Methyl Ether) and their Derivatives

J.C.S., 3721-3726 (Nov. 1955)

Electronic spectra of the title substances are described and analysed for a series of solvents. A qualitative interpretation of the electronic transitions responsible for B-bands suggests that they are of the same type as those causing the K-bands, but also correspond to electronic migrations along the conjugated system formed by all three benzene double bonds. Substituents such as OH, NH, and SH orthe to double bond groups become terminal groups in the absorbing system and responsible for appreciable red shifts, whereas in the para position they are in a side-chain and have much smaller effects. Since the transition moment is much smaller or even negligible, the lower intensities of the B-bands compared with those of the K-bands are accounted for.

III— Tautomeric Equilibria of 2-Nitrosophenols

Ibid., 3727-3733

The electronic spectra of 2-nitrosophenol, 5-methoxy-2-nitrosophenol, and 5-dimethylamino-2-nitrosophenol in various salts are determined and compared with those of the corresponding 2-nitrosoanisoles and 6-methoxy-iminocyclohexa-2:4-dienones. It is shown that these compounds contain an internal hydrogen bond and that, in spite of it, the methoxy and dimethylamino deriv. exist as solvent-dependent tautomeric equilibria. This confirms that an internal hydrogen bond cannot be interpreted as a non-localised bond. In all solvents, the parent epd. is observed only as the true 2-nitrosophenol tautomer. The participation of the nitroso group in an internal hydrogen bond results in a storage displacement of the hydrogen bond results in a strong displacement of the R-band to shorter wavelengths (800–1200 a.). H.H.H.

[4 - Methyl - and 4:8 - Dimethyl - 1:5 - hydroxy naphthalene]

Ng. Ph. Buu-Hoi and D. Lavit

J. Org. Chem., 20, 1191-1196 (Sept. 1955) Both epd., which are best prepared starting with the condensation of 1:5-dimethoxynaphthalene with N-methylformanilide, oxidise rapidly in air. H.E.N. methylformanilide, oxidise rapidly in air.

NNN'N'-Tetraethyl-p-phenylenediamine as Detector of Short-lived Organic Radicals in Solution V. Franzen

Chem. Ber., 88, 1697-1703 (Nov. 1955)

Synthesis of 4-Phenoxycatechol and 2-Phenoxy-

D. E. Janssen, J. VanAllan, and C. V. Wilson J. Org. Chem., 20, 1326-1329 (Oct. 1955)

Preparation and Reduction of N-Methyl-1-β-aminoethyl-2-methoxynaphthalene C. F. Koelsch and H. E. Hood

J. Org. Chem., 20, 1282-1287 (Sept. 1955)

Studies in Light Absorption XI-Substituted Benzaldehydes, Acetophenones, A. Braude and F. Sondheimer

J.C.S., 3754-3766 (Nov. 1955)

- Effects of Steric Conformation on the Ultraviolet and Infrared Spectra of Alicyclic Ethylenic

E. A. Braude and C. J. Timmons

Ibid., 3766-3772

XIII-Steric Effects in ortho-Substituted Styryl and Related Derivatives

E. A. Braude and F. Sondheimer

Ibid., 3773-3776

XIV-Steric Effects in ortho-Substituted Diphenyls E. A. Braude and W. F. Forbes

Ibid., 3776-3782

Aryl-2-halogenoalkylamines. XV—Some Cationic and Basically Substituted Aryl Compounds. F. Bergel, J. L. Everett, J. J. Roberts, and W. C. J. Ross J.C.S., 3835–3839 (Nov. 1955)

The work described in Part XII (J.C.S., 2386 (1953)) is continued and the preparation is recorded of some p-di-(2-chloroethyl)aminobenzene deriv. with side-chains containing basic and cationic groups, e.g. of general formulae (Cl·CH₂·CH₂)₂N·C₄H₄·[CH₂]_n·NRR' and (Cl·CH₃·CH₃)₄N·C₄H₄·[CH₂]_n·N⁴(CH₃)₄N·C₄H₄·[CH₂]_n·N⁴(CH₃)₃N·C₄H₄·[CH₃]_n·N⁴(CH₃)₃N·C₄H₄·[CH₃]_n·N⁴(CH₃)₃N·C₄H₄·[CH₃]_n·N⁴(CH₃)₃N·C₄H₄·[CH₃]_n·N⁴(CH₃)₃N·C₄H₄·[CH₃]_n·N⁴(CH₃)₃N·C₄H₄·[CH₃]_n·N⁴(CH₃)₃N·C₄H₄·[CH₃]_n·N⁴(CH₃)₃N·C₄H₄·[CH₃]_n·N⁴(CH₃)₃N·C₄H₄·[CH₃]_n·N⁴(CH₃)₃N·C₄H₄·[CH₃]_n·N⁴(CH₃)₃N·C₄H₄·[CH₃]_n·N⁴(CH₃)₃N·C₄H₄·[CH₃]_n·N⁴(CH₃)₃N·C₄H₄·[CH₃]_n·N⁴(CH₃)₃N·C₄H₄·[CH₃]_n·N⁴(CH₃)₃N·C₄H₄·[CH₃]_n·N⁴(CH₃)₃N·C₄H₄·[CH₃]_n·N⁴(CH₃)₃N·C₄·[CH₃]_n·N⁴(CH₃)₃N·C₄·[CH₃]_n·N⁴(CH₃)₃N·C₄·[CH₃]_n·N⁴(CH₃)₃N·C₄·[CH₃]_n·N⁴(CH₃)₃N·C₄·[CH₃]_n·N⁴(CH₃)₃N·C₄·[CH₃]_n·N⁴(CH₃)₃N·C₄·[CH₃]_n·N⁴(CH₃)₃N·C₄·[CH₃]_n·N⁴(CH₃)₃N·C₄·[CH₃]_n·N⁴(CH₃)₃N·C₄·[CH₃]_n·N⁴(CH₃)₃N·C₄·[CH₃]_n·N⁴(CH₃)₃N·C₄·[CH₃]_n·N⁴(CH₃)₃N·C₄·[CH₃]_n·N⁴(CH₃)₃N·C₄·[CH₃]_n·N⁴(CH₃)₃N·C₄·[CH₃]_n·N⁴(CH₃)₃N·C₄·[CH₃]_n·[CH₃]_n·N⁴(CH₃)₃N·C₄·[CH₃]_n·N⁴(CH₃)₃N·C₄·[CH₃]_n·[C

Synthesis and Spectral Study of Malonic Anils A. Van Dormael and J. Nys

XXVIII Congrès International de Chimie Industrielle Chim. et Ind., 74, 180 (Oct. 1955)
The synthesis is described of malonic anils substituted

by electrophilic or nucleophilic groups, e.g. NO_3 , COOH, $COOC_2H_3$, $CO\cdot CH_3$, $O\cdot CH_3$, by condensation of the acetal of propargylic aldehyde with the corresponding anilines in presence of HCl. During chromatography on alumina, the HCl is removed, leaving the corresponding s in a pure state.

The optical properties of these colouring matters have been determined in neutral and acid solution. From the absorption maxima of the acid solutions and those of the monomethin cyanine derivatives of the benzothiazole, the authors have been able to calculate the theoretical absorption maximum of the anilinovinylsubstituted salts (Idem, Bull. Soc. Chim. belg., 61, 614 (1952)). The disagreement with the law of averages observed for the anilinovinyl derivatives is discussed.

Reactions with 16N. XIX-Constitution of Potassium Phenylantidiazonte

K. Clusius and H. Hürzeler

Helv. Chim. Acta, 38, 1831-1833 (Dec. 1955) Aniline (I) is converted successively into benzene-Anime (I) is converted successively into behaviorable diazonium chloride (II), potassium phenylantidiazonte (III), phenylhydrazine, and into aniline (IV) and ammonia (V). Almost all the ¹²N appears in IV when ¹²N-aniline is used as I or in V when Na¹⁵NO₃ is employed. In spite of the step II -> III requiring 50-75% KOH at 140 c., no interchange of nitrogen atoms can be occurring. These expet do not decide whether the sun anti-impression is expt. do not decide whether the syn-anti isomerism is H.E.N. steric or structural.

Kinetics and Mechanism of Diazotisation-VIII

Mh. Chem., 86, 668-671 (Aug. 1955) The small free energy of activation, 4.7 kcal., and the small enthalpy of activation, 4.6 kcal., for the diazotisation of unionised aniline by nitrosyl chloride are attributed to the temporary attractive forces (electromeric and dispersion) exercised by the aniline. H.E.N.

Mechanism of Coupling H. Zollinger

IX-Kinetic Effects of Deuterium Substitution

and Basic Catalysis

Helv. Chim. Acta., 38, 1597-1616 (Oct. 1955)

The reaction (I) of p-chlorobenzenediazonium chloride with G acid shows a salt effect which is smaller than that theoretically expected. This reaction is also strongly catalysed by pyridine. Hodgson and Marsden (J.S.D.C., 60, 16 (1944)) postulated the equilibrium-

$$Ar \cdot N_s^+ + N \Rightarrow Ar \cdot N \cdot N \cdot N$$
 (a)

as the first step in catalysed coupling reactions. From kinetic measurements on I, the equilibrium constant of (a) is calculated, and hence the expected equilibrium conen. of the azopyridinium ions derived. The experimentally determined values of the latter are much smaller, and Hodgson's mechanism cannot apply.

When I-D-2-naphthol-6:8-disulphonic acid is used, the velocity of I is reduced to less than one-sixth. This is the first example of an aromatic electrophilic substitution which exhibits a kinetic isotope effect the magnitude of which corresponds to that expected theoretically. The reaction of o-methoxybenzenediazonium chloride \rightarrow NW acid or 2-D-1-naphthol-4-sulphonic acid, which is not base-catalysed, shows no kinetic isotope effect. All kinetic data are in agreement with the mechanism-

$$\mathbf{Ar} \cdot \hat{\mathbf{N}}_{1} + \mathbf{R} \cdot \mathbf{H} \stackrel{k_{1}}{\rightleftharpoons} \mathbf{H} \cdot \hat{\mathbf{R}} \cdot \mathbf{N} \cdot \mathbf{N} \cdot \mathbf{Ar}$$
 (1)

$$\mathbf{H} \cdot \mathbf{\hat{R}} \cdot \mathbf{N} \cdot \mathbf{N} \cdot \mathbf{Ar} + \mathbf{B} \stackrel{\mathbf{\hat{r}_2}}{\rightarrow} \mathbf{R} \cdot \mathbf{N} \cdot \mathbf{N} \cdot \mathbf{Ar} + \mathbf{H} \stackrel{\mathbf{\hat{r}}}{\rightarrow}$$
 (2)

where R.H is the naphtholate ion and B the base, such as H_1O , OH^- , or pyridine, and each k is the appropriate rate constant. It is possible to measure k_1 and the ratio k_2/k_{-1} . but not k_1 and k_{-1} themselves. The reaction is base-catalysed and shows isotope effects if k_3/k_{-1} is small. This is the only condition; isotope effects do not prove that k, is small. Reactions without catalysis or isotope effects proceed by the same mechanism. The species $H \cdot \hat{R} \cdot N \cdot N \cdot A_{F}$ is a thermodynamically metastable intermediate and not a transition state.

X-Dependence of the Kinetic Isotope Effect on Base Concentration and on the Diazo Component Ibid., 1617-1622

As expected on the basis of the mechanism postulated, although the overall isotope effect for I decreases with increasing conen. of base, the isotope effect for stage (2) remains constant. As increasing acidity in the diazonium epd. would help elimination of the proton, k_1 and k_2/k_- would thereby be increased and a decreased overall isotope effect expected. This is found on comparing three diazo components. Strong support is thus adduced for the mechanism advanced.

XI- The Nature of the Proton Elimination

In comparing the catalytic effect of different bases on I. it is found that pyridine and 3- and 4-picoline behave as expected from their strengths, but catalysis by 2-picoline and 2:6-lutidine is only one-third and one-tenth as much respectively. This is readily understandable if stage (2) proceeds via a transition complex, but not if it takes place by dissociation of the proton followed by its reaction with the base. Base-catalysed coupling reactions may exhibit catalysis by OH-, i.e. a sudden increase in coupling velocity above pH 11.

H.E.N.

Sulphonyl Chlorides of Azo Dyes

M. Schmid and R. Mory Helv. Chim. Acta, 38, 1329-1338 (Oct. 1955)

Such acid chlorides are required as intermediates in the preparation of amides which may be useful as pigments, of, the Chromophthal range (Ciba). c-Hydroxyazosulphonic acids are found to be convertible into their chlorides by means of PCl, in chlorobenzene or dioxan or by POCl, The phenolic group is not attacked at or below room temp. If the hydroxyl group has substituents in both the positions ortho to it, higher temp. may be used. Several examples, including pyrazolone (I) and acetoacetarylides (II), are given. In the case of oo-dihydroxyazo dyes the hydroxyl groups must be protected, usually by benzoylation, though acetylation is occasionally employed. The enol groups of I and II are not acylated. Derivatives of 1-amino-2naphthol-4-sulphonamide are obtained by these methods, and these cannot be made, or can be prepared only with great difficulty, by the direct route. In some cases, in the conversion of the sulphonyl chloride into the sulphonamide deacylation occurs simultaneously. The present work is covered e.g. by FP 1,073,234. The paper is illustrated by colour photomicrographs.

Synthesis of some Aromatic Amines containing Trifluoromethyl Groups and their Use in Azoic

Dycing
A. E. Porai-Koshits, B. A. Porai-Koshits, L. S. Éfros,
M. I. Krýlova, D. A. Livshits, K. Yu. Mar'yanovskaya,
I. P. Aleksandrova, and K. É. Ul'man

J. Appl. Chem. U.S.S.R., 28, 969-975 (Sept. 1955) The main purpose of the investigation is the study of the synthesis of trifluoromethyl (TFM) deriv. of aromatic amines that have been described as suitable azoic components in the patent literature. The syntheses of the following cpd. are described—3 TFM., 2-chloro-5-TFM., 4-chloro-2-TFM., 3-nitro-5-TFM., 2-nitro-4-TFM., and 2-methoxy-5-TFM-anilines; 5-TFM-m-phenylenediamine; the 3-TFM-, 2-chloro-5-TFM-, and 2-methoxy-5-TFManilides of 3-hydroxy-2-naphthoic and acetoacetic acids; and the 4-chloro-2-TFM-anilide of acetoacetic acid. No details of the properties of the dyeings produced by these components are given, but it is stated that 2-chloro-5-TFM-, 4-chloro-2-TFM-, and 2-methoxy-5-TFM-anilines, used as diazo components in azoic dyeing, give dyeings of exceptional brightness and fastness, and that the arylexceptional brightness and restriction, and around dyeing amides also are of practical interest in azoic dyeing.

A.E.S.

Physicochemical Properties of Hydrophobic Dyes E. A. Veller and B. A. Poraï-Koshits

II- Absorption Spectra in various Organic Solvents

J. Appl. Chem. U.S.S.R., 28, 750-755 (July 1955) Absorption spectrum measurements previously reported (see j.s.p.c., 71, 553 (Sept. 1955)) are extended to soln. of the same and other oil-sol. and disperse dyes (I, II, IV— VII—see below) in other solvents (ethanol, acctone, benzene, dibutyl phthalate). In general, the effects of the variation of the solvent on the position of the absorption max. and the value of the molecular extinction coefficient are only slight.

III - Solubility of Dyes in Organic Solvents

Ibid., 28, 857-863 (Aug. 1955) Saturated soln. of dyes are prepared in the cold (exact temp. not given), and the conen. is determined colorimetrically after suitable dilution, the calculations being based, in part, on assumptions shown to be valid in previous investigations (see above). The solubilities (% by wt.) of each dye are given below, the solvents being, respectively, ethanol, acetone, dichloroethane, benzene, and dibutyl phthalate—Sudan Yellow G (Yellow G fatsoluble) (I), 0-038, 0-304, 2-84, 3-04, 0-9; Scarlet G for acetate rayon (II), 0-28, 2-16, 1-15, 0-5, 0-96; Blue K for acetate rayon (III), 0.12, 0.24, 0.19, 0.04, 0.12; Sudan Red 7B (IV), 0.046, 1.5, 12.7, 15.0, 3.4; Cyanine Green 5G (1:4-dihydroxy-5:8-di-p-toluidinoanthraquinone), 0.028, 0.17, 0.81, 0.8, 0.43; Sudan Blue G (VI), (1-methylamino-4-p-toluidinoanthraquinone), 0-046, 0-19, 1-28, 0-9, 0-0528; Acid Brilliant Green G (VII) (tetraethyl analogue of C.I. 735), 10-5, 0-19, 0-004, 0, 0 (for constitutions of I-IV see J.S.D.C., 71, 553 (Sept. 1955)). A.E.S.

Geometrical Isomerism of the Azonaphthalenes M. Frankel, R. Wolovsky, and E. Fischer

J.C.S., 3441-3445 (Oct. 1955) cis-2:2'- and cis-1:2'-Azonaphthalene have been pre pared by ultraviolet irradiation of the solutions of the corresponding trans isomers, isolated by chromatography on alumina at low temp., and characterised by their absorption spectra; cis-1:1'-azonaphthalene has been similarly prepared and characterised in solution containing up to 60% of cis-isomer, but it was not isolated. A paper-chromatographic technique, analogous to that used for azobenzene (Frankel and Wolovsky, Experientia, 10, 356 (1954)), is also serviceable for the micro-separation and -estimation of the two isomers of 2:2'-azonaphthalene.

Metabolism of Radioactive Fast Yellow K. Karrer, E. Broda, R. Stark, O. Hromatka, and W. Zischka

Mh. Chem., 86, 444-448 (June 1955) Sodium 4-aminoazobenzene-3:4'-disulphonate tagged with radioactive C at position I' was fed to rats and it found that qualitatively the activity distributed itself amongst the organs as it did with radioactive Butter Yellow, though absorption and elimination (mainly through the urine) were quicker. The different solubilities of these two dyes cannot be the explanation of the difference in their carcinogenic power, the cause of which must lie in the details of their structures. H.E.N.

Polarisation of Azobenzene as a Vapour H. C. Freeman, R. J. W. Le Fèvre, D. A. A. S. N. Rao, and I. G. Ross

J.C.S., 3840-3845 (Nov. 1955) Apparatus for taking polarisation measurements up to 340°c. has been used for gaseous azobenzene. The total polarisation of the latter is found to be ca. 66 c.c., i.e. of the order to be expected from the known mol. refraction, indicating the atomic polarisation to be ~0 e.c. H.H.H.

o-Mercaptoazo Compounds. VII—Preparation and Debenzylation of 2:2'-Dibenzylthioazobenzene A. Burawoy, A. Chaudhuri, and C. E. Vellins

J.C.S., 3798-3804 (Nov. 1955) 2:2'-Dibenzylthioazobenzene (I), when treated with bromine in glacial acetic acid, gives 2-benzylthicazo-benzene-2'-sulphenyl bromide and tribromide, di-[o-(o'-bromothiophenylazo)phenyl] disulphide, and azoto normalize the state of the s benzene in benzene ether soln.

p-Nitrobenzoylacetic Acid and its Derivatives in relation to Azoic Coupling Components
C. Coll, A. Milá. and J. Pascual Vila

XXVIII Congrès International de Chimie Industrielle Chim. et Ind., 74, 178 (Oct. 1955)

The nitro group of the ethyl ester has been reduced to amino, or the ester group has been converted to anilide, or both processes together. The products are potential first components, and have been coupled with diazotised 8.R.C. m-nitroaniline.

pH Indicators H. Ikegami and H. Hiyama

VII- Phenylazo-1-naphthol Derivatives

Science & Industry (Japan), 27, 328–330 (1953)

Chem. Abs., 49, 12179 (25 Sept. 1955)

In phenyl-substituted derivatives of phenylazo-1naphthol (o-, m-, and p-CH₂), CH₂O, Cl, NO₂, COOH;
o-C₂H₂O; p-Br; 2:4:5-tribromo; 2:4-dinitro), mostly
changing colour on the alkaline side, those having a positive radical in the benzene nucleus (azo-dominant type) show relatively small pK values and change colour indistinctly, while those having a negative radical, particularly NO₂ (hydrazone-dominant type), show large pK values and deep distinct colours on the alkaline side, except for the COOH derivatives.

VIII—Mechanism of Colour Change of Hydroxy-azo pH Indicators

Ibid., 27, 330-334 (1953) The absorption spectra of azo-hydrazone tautomeric C₄H₄·N:N·C₁₆H₄OH derivatives show that those azo-dominant at pH 7-0 shift to the hydrazone-dominant type if the pH is increased. The NO₃ derivatives which are hydrazone-dominant at pH 7-0 shift to the azinitro-dominant structure at higher pH, thus confirming Hantssch's theory of nitro-azinitro tautomerism.

X—Effect of Chlorine and Alkoxyl Groups on Phenylazonaphtholsulphonic Acids

1bid., 28, 16-21 (1954)
Single substitution of Cl, CH₂O or C₂H₃O in the diazonaphtholsulphonic Acids

component (aniline) gave stable compounds, but further substitution of NO₂ gave good alkaline indicators. The best indicator was obtained with NO₂ in para position in aniline with Cl, CH₂O or C₂H₂O substituted in the orthoposition. When NO₂ was orthogod by Cl, CH₂O or CH₂O position. When NO2 was ortho and the Cl, CH2O or C2H4O group para the change in colour was indistinct.

XI-Effect of the Sulphonic Group in Phenylazonaphthol

Ibid., 28, 22-24 (1954) Introduction of SO₂H into the diazo component yielded compounds more soluble in water, intensified the effect of NO_a substitution, and deepened the colour change on the alkaline side. In some cases, however, the pK was lowered. C.O.C.

Aromatic Diazo Compounds
XVII—Dark Brown Direct Cotton Dyes of Superior Light Fastness

J. Poskočil and Z. J. Allan

Collection Czechoslov. Chem. Commun., 20, 609-614 (1955)

XVIII- New Red and Violet Direct Cotton Dyes derived from Benzotriazole F. Mužik and Z. J. Allan Ibid., 615-622

Chemistry of Heterocyclics. XXIX and XXX—Diaromatics in the Thiophen Series
G. N. Jean and F. F. Nord
J. Org. Chem., 20, 1363-1369; 1370-1378 (Oct. 1955)

Three 2-phenylthiophens, three 2:2'-dithienyls, and two 3:3'-dithienyls, all carrying bulky substituents in the ortho positions, are prepared. Their ultraviolet absorption spectra when compared with those of the uncoupled parts showed that the 2:2'-dithienyls can achieve coplanarity

Environmental Influences and the Colour of Some meroCyanines
Y. Hirshberg, E. B. Knott, and E. Fischer

whilst the others cannot.

J.C.S., 3313-3321 (Oct. 1955)

The varied behaviour of a series of dimethinmero cyanine dyes with respect to their absorption spectra is to be attributed, in general, to differences in their polarity. The more highly polar dyes exhibit two absorption bands in alcohols or aq. alcohol, the long-wavelength band being attributed to the dye itself and the shorter-wavelength band to a quaternary cation formed between the solute and the solvent. The effects are examined of certain environmental factors on the absorption spectra, e.g. the degree of dilution, particularly in alcohols, the partial replacement of solvent ethanol by water, and temper H.H.H.

Monomethin Cyanines- A New Reaction

J. Nys
XXVIII Congrès International de Chimie Industrielle
Lud 74, 178-180 (Oct. 1955)

Quaternary salts of heterocyclic bases of the azole class possessing a reactive methylthic group in the 2-position when acted upon by alkyl or arylalkyl acid anhydrides yield the corresponding symmetrical monomethin cyanines carrying the alkyl or aryl group of the anhydride less e.g. one methyl, ethyl, or phenyl group. A high yield is obtained, The mechanism is discussed. S.R.C.

Pyrylocyanine Dyes

XXVIII Congrès International de Chimie Industrielle

Chim. et Ind., 74, 180 (Oct. 1955) Monomethin pyrylocyanines are obtained by condensation of tetrahydroxanthylium tetrachloroferrate with aromatic or heterocyclic aldehydes. Optical properties of the colouring matters in solution and the relation between constitution and colour are discussed. S.R.C.

Cyanine Dyes . Zenno

XIII- Conversions of Parent Compounds among

Various Cyanine Dyes

J. Pharm. Soc. Japan, 74, 1236-1240 (1954)

Chem. Abs., 49, 14736-7 (10 Nov. 1955)

XIV—Syntheses of Styryl Dye Analogues con-

taining Halogen Atoms Ibid., 1241-1243

Organic Fluorescent and Photosensitive Substances

Rept. Inst. Ind. Sci. Univ. Tokyo, 4, 93-151 (1955)
Chem. Abs., 49, 14545 (10 Nov. 1955)
Review of the fluorescence and photochemistry of dyes
and other organic compounds. 176 references. C.O.C.

Cyclisation of 1:4- and 1:5-Di-(α-anthraquinonyl-amino)anthraquinones to Derivatives of Carbazole W. Bradley and P. N. Pandit

J.C.S., 3399-3405 (Oct. 1955) When heated with AlCl_a, TiCl₄, or KOH, 1:4-di-(a-anthraquinonylamino)anthraquinone (I) cyclises to the related hendecacyclic triquinone (II). The 1:5 isomer of I behaves analogously. II also results when 6-(a-anthraquinonylamino)dinaphtho(2:3-a,2':3'-i)-carbazole-5:10:15:17-diquinone is heated with TiCl₄, the second carbazole nucleus forming more readily than the first. An isomeric triquinone is analogously formed from the 1:5 isomer.

H.H.H.

Synthesis of 1-, 2-, 3-, 4-, and 9-Chloromethyl-

phenanthrene P. J. C. Fierens, R. H. Martín, and J. Van Rysselberg Helv. Chim. Acta, 38, 2005-2008 (Dec. 1955)

Kinetic Studies on Derivatives of Polycyclic Hydrocarbons

I-Solvolysis and Iodine-exchange Reaction of Chloromethyl Compounds

P. J. C. Fierens, H. Hannaert, J. Van Rysselberge, and R. H. Martin

Helv. Chim. Acta, 38, 2009-2021 (Dec. 1955)

II — Alkaline Hydrolysis of Carboxylic Esters M. Adam-Briers, P. J. C. Fierens, and R. H. Martin Ibid., 2021-2026

o-Dicarboxylic Acid Derivatives of Pyrazole, iso-Oxazole, and Pyrimidine R. G. Jones and C. W. Whitehead

J. Org. Chem., 20, 1342-1347 (Oct. 1955) Diethyl ethoxymethyleneoxalacetate, C.H.O.CO.CO. C(:CH-OC₂H₃)·CO-OC₂H₃ (I), reacts with hydrazine to give diethyl pyrazole-4:5-dicarboxylate, with phenylhydrazine to give the corresponding 1-phenyl cpd., and with hydroxylamine to give diethyl isooxazole-4:5-dihydroxylamine to give diethyl isooxazole-4:5-di-carboxylate. With urea, I exchanges the ethoxymethylene group for ureidomethylene, but when further heated near its m.p. (about 170°c.), it yields diethyl 2-hydroxypyrimidine-4:5-dicarboxylate.

Polynuclear Compounds. I- Synthesis of Pyrene

J. Org. Chem., 20, 1392-1395 (Oct. 1955) See J.B.D.C., 71, 154 (1954). H.E.N.

Di- and Poly-cyclic Azulenes. XX-Azulenes of Lignite Tar

Treibs and K. Krumbholz Annalen, 595, 208-211 (Oct. 1955) Azulene and its 3:8-dimethyl-5-isopropyl and

dimethyl deriv. are isolated. H.E.N. Synthesis of 10:11-Dimethyl-1:2-benzazulene and 2:7-Dimethylindeno-1':2'-9:10-phenanthrene

Chem. Ber., 88, 1787-1792 (Nov. 1955) Fluorene-2-diazonium chloride couples instantly with the former hydrocarbon in position 3 to change the the former hydrocarbon in position of colour from blue to red-brown, which becomes deep blue H.E.N.

Synthesis and Properties of Fluorene-1:4:9-trione and other Fluorenone Derivatives C. F. Koelsch and R. N. Flesch

J. Org. Chem., 20, 1270-1276 (Sept. 1955)

Synthesis of 5:6-Benzopyrrocoline

E. M. Roberts, M. Gates, and V. Boekelheide J. Org. Chem., 20, 1443-1447 (Oct. 1955) Quinaldine is condensed with ethyl oxalate and reduced by sodium borohydride to give, unexpectedly, 1-(2-quinolyl)propane-2:3-diol. Treatment with HBr and subsequent steam-distillation from alkali complete the

2-Acetylamino-5-hydroxyfluorene E. K. and J. H. Weisburger

W. Ziegenbein

J. Org. Chem., 20, 1396-1401 (Oct. 1955) This is prepared from fluorenone-4-carboxylic acid and is shown to be a metabolite of 2-acetylaminofluorene by carrier isotope dilution experiments. H.E.N.

Heterocyclic Imines and Amines IV—Imidines from 3:4:5:6-Tetrahydrophthalic Acid and cis-Hexahydrophthalic Acid. An Unusual Dehydrogenation

G. E. Ficken and R. P. Linstead J.C.S., 3525-3529 (Oct. 1955) The preparations of the first amidine of the maleic acid series, 3:4:5:6-tetrahydrophthalimidine, and of the related cis-hexahydrophthalimidine (I) are reported, together with a preliminary account of their easy conversion into tetrazaporphin pigments. Reaction of I with aniline in boiling ethanol does not give the expected diphenylimine but its dehydrogenation product—a most unusual reaction, which has been repeated several times.

Nickel-Formazyl

V—The Imidine of αα'-Dimethylsuccinic Acid R. P. Linstead and M. Whalley Ibid., 3530-3536 (Oct. 1955)

DL-aa'-Dimethylsuccinonitrile reacts with NH₃ in methanol at 150°c, to give DL-2:5-di-imino-3:4-dimethylspyrrolidine (dimethylsuccinimidine) (I), which becomes blue at ~130°c. The latter reacts normally with hydroxylamine hydrochloride to give a dihydroxyminopyrrolidine, but abnormally with other primary amines: e.g. with aniline dehydrogenation to the dimethylmaleic deriv. occurs; aniline hydrochloride gives the phenylimino-anilide; and benzylamine and cyclohexylamine give substituted imino-imides.

н.н.н.

Conjugated Macrocycles

XXVI—Octamethyltetrazaporphin
M. E. Baguley, H. France, R. P. Linstead, and M. Whalley

J.C.S., 3521–3525 (Oct. 1955)
The reaction of dimethylanelei and dimethylamaronitriles with various organomagnesium compounds gives magnesium octamethylaterazaporphin (II: M = Mg). Removal of Mg from II gives the metal-free pigment (I), which has been converted into the pigments II: M = Cu, Ni, Co, or Zn.

XXVII—Formation of Tetrazaporphins from Imidines. Tribenzotetrazaporphin J. A. Elvidge and R. P. Linstead

Ibid., 3536-3544 (Oct. 1955) Condensations between imidines (with elimination of NH_a) are discussed. Self-condensation of 4 mol. of either a saturated or an unsaturated (or aromatic) imidine can yield only compounds containing the tetrazaporphin skeleton at unfavourable hydrogenation levels, and so can afford tetrazaporphins only through supplementary oxidation-reduction processes. The condensation between imidines at different hydrogenation levels, however, can yield directly tetrazaporphins or their di- or tetra-hydro deriv. The preferred reaction is between 3 mol. of un-saturated and 1 mol. of saturated imidine. Metal-free phthalocyanine is formed by heating di-iminoisoindoline, but in appreciable yields only with hydrogen donors gives the metal phthalocyanines under mild conditions. present. Heating the imidine with metal salts or metals indoline (in the absence of metals) gives a mixture of tetrazaporphin pigments, containing mainly phthalocyanine and a new blue compound, tribenzotetrazaporphin, isolated by chromatography. H.H.H.

Chlorophyll and Related Substances U. Eisner and R. P. Linstead

J.C.S., 3742-3749 (Nov. 1955)

I—Synthesis of Chlorin Chlorin (1), the parent substance of chlorophyll, has been synthesised from 2-dimethylaminomethylpyrrole (II) in one step by heating it with 1 equiv. of C_2H_2MgBr in boiling xylene, when magnesium chlorin is produced as a greenishblue solution with an intense red fluorescence; this is immediately converted into the stable metal-free epd. by cold dil. HCl (in yields up \$\tilde{c}0.3.9\%). Cu and Mg deriv. have been prepared, and light absorption data are given.

II— Dehydrogenation of Chlorin to Porphin and the Number of Extra Hydrogen Atoms in the Chlorins Ibid., 3749-3754

Dehydrogenation of chlorin by quinones of high potential (e.g. tetrachloro-1:2-benzoquinone and 2:3-dichloro-5:6-dicyano-1:4-benzoquinone) rapidly converts it to porphin. The stoicheiometry of this reaction is studied, and chlorin shown to be a dihydroporphin. H.H.H.

Unsaturated

Coordinatively Complexes M. Seyhan

Mh. Chem., 86, 545-548 (June 1955)

Flushing Process for Pigments

L. Korfhage

Fette, Seifen, Anstrichmittel, 56, 119-120 (1954)
Chem. Abs., 49, 14339 (25 Oct. 1955)
Review, 17 references. C.O.C.

Light-sensitive Diazonium Compounds for use in

Diazotype Printing
E. N. Mason & Sons
BP 740,565

The diagonium compounds derived from amines of formula—

(Alk = $\mathrm{CH_3}$, $\mathrm{C_2H_5}$, $\mathrm{C_2H_7}$ or $\mathrm{C_4H_9}$; n=3-10) are very sensitive to light, show good coupling energy, give dark colours and do not develop a mercaptan odour when decomposed by light.

Blue to Violet Nitro Hair Dyes

Unilever BP 741,334 Compounds of formula $(NO_2)_mR^1(NHR^2)_n(NR^3R^4)_p$ $(R^1 = \text{aromatic}; R^2, R^3 \text{ and } R^4 = \text{aliphatic containing at least one OH group; } m, n \text{ and } p = \text{whole numbers}, e.g. 1-(di-<math>\beta$ -ethylolamino)-4- β -ethylolamino-3-nitrobenzene, dye animal fibres deep blue to violet. Used on grey human hair they suppress the reddish tints which other nitro dyes tend to produce. C.O.C.

Red Monoazo Acid Dyes

Aryl esters of orthanilic acid-

(R = benzene or naphthalene series aryl which may contain non-salt-forming substituents) are diazotised and acid-coupled with y-acid to give bright bluish red acid dyes of good light fastness. If R = phenyl the dyes have good levelling properties, and if R contains as substituents Alk or cycloalkyl of > 3 C, the resultant dyes have good wet fastness. Thus o-nitrobenzenesulphonyl chloride is reacted with sodium phenate, and then the nitro group is reduced to amino. The phenyl ester of orthanilic acid so produced is dissolved in 80% acotic acid, diazotised with NaNO₈ and HCl at 10°c., and coupled with y acid in presence of acetic acid to give—

which dyes wool level bluish red.

E.8.

Monoazo Acid Dyes having a β-Chloroethylsulphone Group

FH BP 740,533 Diazo compounds of amines having a grouping—SO₃-CHR-CH₂Cl (R = H, Alk or Hal) are coupled with suitable coupling components to give acid dyes. The dyeings or prints produced on animal, polyamide, cellulose or cellulose acctate fibres by these dyes are treated with weak alkalis, which fix the dye probably by converting this grouping into a reactive vinylsulphone group which combines with reactive sites in the fibre. The diazo components may be made by reacting a suitable sulphinic acid, with β -chloroethanol or ethylene oxide, followed by replacing the OH group by Cl by treatment with SOCl₃ or HCl. Thus, the monoazo compound 1-amino-2-anisole-5- β -chloroethylsulphone \rightarrow 1-naphthol-5-sulphonic acid, is an acid dye for wool which when aftertreated with 1% soap solution gives a red of good wet fastness properties.

Grey Metal(Chromium)-complex Monoazo Dyes for Wool, Nylon, etc.

ICI

The chromium complexes containing 1 atom of Cr to 2 mol. of oo'-dihydroxymonoazo compounds—

(R = Alk of 2-4 C; X = Alk of 1-4 C) are grey dyes for wool and nylon and exhaust better from a neutral bath than the similar dyes of BP 667,168 (J.S.D.C., 68, 190 (1952)) in which R = CH₂. Thus the monoaco compound 2-aminophenol-4-ethylsulphone \rightarrow 1-earboethoxyamino-7-naphthol is boiled in aqueous ethylene glycol with basic chromium acetate. The product dyes wool and nylon or mixtures of these fibres bluish grey from a neutral or acetic acid bath. E.S.

Metal(Chromium and Cobalt)-complex Monoazo Dyes for Wool and Nylon Ciba

The Cr and Co complexes of the dyes obtained by coupling a diazotised o-aminophenol (preferably containing a sulphamyl substituent) with a 1-acylamino-7-naphthol, followed by complete or partial hydrolysis of the acylamino group, and containing less than 1 atom of metal to 1 mol. of monoazo compound dye wool and nylon from neutral or weakly acid baths. The acylamino group may be e.g. acetylamino or a carbamic methyl (or ethyl) ester group. Purer dyes and better yields are obtained than by using the 1-amino-7-naphthol itself as the coupling component. Thus 2-aminophenol-4-sulphonmethylamide is diazotised and coupled with N-acetyl-1-amino-7-naphthol. The monoazo compound so produced is boiled for 6 hr. with aq. NaOH and sodium chromosalicylate so that both hydrolysis and metallisation take place. The product dyes wool green-grey from a weakly alkaline, neutral, or weakly acid bath.

E.S.

Metal-complex Monoazo Dyes for Wool

Ciba BP 741,602
The metal (especially Cr and Co) complexes containing 1 atom metal to 2 mol. of a monoazo compound free of SO₂H and COOH groups—

(R¹ = a benzene residue in which OH is ortho to the azo link; R³ = a benzene residue; at least one Alk or Hal substituent is present in R¹ or R²), or to 1 mol. of such a monoaxo compound and 1 mol. of an oo'-dihydroxy-benzeneazonaphthalene compound free of SO₂H and COOH groups, are water-soluble and dye wool from a neutral or weakly acid bath. Thus 2-aminophenol-4-sulphonamide is diazotised and coupled with 1-p-chlorophenyl-3-methyl-5-pyrazolone, and the insoluble monoazo

compound so formed is boiled with sedium potassium chromosalicylate and NaOH, and the soluble dye isolated by evaporation in vacuo. It dyes wool pure scarlet from a neutral or acetic acid bath. E.S.

Metal(Chromium)-complex Monoazo Dyes for Wool and Nylon containing a Hydroxyalkoxy Group BP 740,589

The chromium complexes derived from monoaso dyes free of acid water solubilising groups but containing an hydroxyalkyl group—

(X = H, or a non-salt forming group, in particular Hal; R^1 = H or subst. or unsubst. Alk of 1 or 2 C; R^2 = hydroxyalkyl; Λ = residue of β -naphthol coupled in the 1-position, and may contain non-salt forming groups), in which 2 monoazo mol, are combined with at least 1 Cr atom, are dyes of good levelling properties on wool and nylon. Their solubility in water may be improved by mixing with alkaline salts or with anionic wetting and dispersing agents. Thus, hydroquinone is hydroxyethylated, mononitrated, and reduced to give 2:5-bishydroxyethoxyaniline, which is diazotised and coupled with β -naphthol. The monoazo compound so formed is heated with chromium acetate and NaOH in ethylene glycol at 145–155°c, for 8 hr. The product after dispersing in soap dyes nylon navy blue from a neutral or weakly acid bath. E.S.

Dischargeable Monoazo Direct Cotton Dyes for Diazotisation and Development on the Fibre

Gy BP 740,862 Direct dyes which after diazotisation and development (e.g. with β -naphthol) on the fibre have excellent wet fastness properties and discharge to a good white are made by diazotising an amino compound—

 $(X=H, Cl, CH_3, OCH_3 \text{ or } 8O_3H; Y=H \text{ or } CH_3; n \text{ and } m=0 \text{ or } 1)$ and coupling with an N-m(or p)-aminobenzoyl or an N-m(or p)-[m'(or p')-aminobenzoyl derivative of J acid, or with a derivative of it having a thiazole or iminazole ring. Thus 5-p-aminobenzoylaminosalicylic acid is condensed with p-nitrobenzoyl chloride and the product reduced with $Na_s 8$, to give 5-p-(p'-aminobenzoylamino)benzoylaminosalicylic acid, which is diazotised and coupled with N-p-aminobenzoyl-J acid. The monoazo compound so formed dyes cellulose yellowish-red, which is diazotised and coupled on the fibre with β -naphthol to give a dischargeable scarlet. E.S.

Blue Metal(Copper)-complex Disazo Direct Cotton Dyes ICI BP 740.570

Copper-complex disazo dyes-

(R = H, benzoyl, or subst. benzoyl) are direct blues whose hue and light fastness are not materially affected by application of a crease-resist finish, e.g. a urea-formaldehyde resin. Thus dianisidine is tetrazotised and coupled first with 1-amino-8-naphthol-4:6-disulphonic acid in presence of soda ash, and then with 2-amino-1-chloro-5-naphthol-7-sulphonic acid in aq. ammoniacal pyridine. De-alkylation and coppering are achieved by heating with ammoniacal copper sulphate. The product is a greenish-navy direct dye.

E.S.

Azoic Dyes which develop by Neutral Steaming Fran BP 740,579

The triazens obtained by condensing N-substituted anthranilic acids—

(R = Alk, aralkyl, or cycloalkyl of > 2 C; X = H or Hal) with diazo compounds derived from amines having a dissociation constant not more than 3.5×10^{-13} couple with azoic coupling components under neutral steaming conditions. Suitable diszotisable amines include dichloroanilines, nitro-anilines, -toluidines, -anisidines, etc. Thus 5-nitro-2-aminotoluene is diazotised and condensed with N-butylanthranilic acid in presence of soda ash, and the triazen formed is salted out, and dried at a moderate temperature. It is mixed with 3-hydroxy-2-naphtho-otoluidide, and a suitably thickened paste of the mixture containing NaOH and thiodiglycol is printed on cotton. Steaming for 7-8 min. in neutral saturated steam develops a bordeaux azoic print. E.8.

Grey Metal-complex Trisazo Direct Cotton Dyes

The Cu and Ni complexes of trisazo dyes Ri-N:N·R³. N:N·R³·N:N·R⁴ (R¹ = residue of diazo component free of OH groups; R² = aryl of <11 C; R³ = aryl of benzene OH groups; R² = aryl of <11 C; R³ = aryl of benzene series having an O-Alk group ortho to the azo link between R³ and R⁴; R⁴ = the residue of a 5-pyrazolone, or of an ortho-coupling naphtholsulphonic acid) are grey direct dyes whose good light fastness is not appreciably diminished by application of a crease-resist finish. Thus the aminomonoazo compound 2-naphthylamine-4:8-disable could be application of a crease-resist finish. acid >1-naphthylamine-7-sulphonic acid is and coupled with p-cresidine. The aminodiazotised and coupled with p-cresidine. The amino-diazo compound so formed is then diazotised and coupled with 1-naphthol-4-sulphonic acid in presence of soda ash and a little pyridine. The salted-out dye is then stirred at 90-95°c. with aq. copper sulphate in presence of ammonia and pyridine to give the complex—

$$\begin{array}{c|c} SO_3H & O-Cu-O \\ \hline \\ SO_3H & N:N & N:N \\ \hline \\ SO_3H & SO_3H \end{array}$$

Blue Azoic Dyes for Acetate Rayon, Nylon, etc. BP 740,555

Azoic blues of good fastness to cross-dyeing are produced on acetate and polyamide fibres by using as diazo components the aminoazo compounds 2:5-dimethoxyanilinea-naphthylamine or 2-amino-5-methoxytoluene → a-naphthylamine, and as coupling components the o-anisidide, the 4-methoxy-2-methylamilide, or the 5-chloro-2:4-dimethyoxyanilide, of 3:2-hydroxynaphthoic acid. Both components are applied together from a single bath under alkaline conditions, the material is then treated in cold nitrous acid, and coupling effected by heating to 60°c. in aq. sodium acetate.

Sodium 4:4'-Bis(3:4-methylenedioxybenzoylamino)stilbene-2:2 disulphonate—Fluorescent Brightening Agents for Cellulosic Materials DuP USP 2,700,046

Compounds of formula-

(n = a small integer, e.g. 1 or 2; M = H or a cation) are excellent fluorescent brightening agents for cellulose USP 2,700,053

Compounds of formula-

(R = methoxy attached in 4 or 5 position; M = H or a cation) are even better than the above.

Triazine Fluorescent Brightening Agents USP 2,700,665 General Aniline

Compounds of formula-

 $(NXY = NH_1 \text{ or the residue of a primary or secondary amine; } Z = COOM \text{ or } SO_3M \text{ } (M = H \text{ or cation); } R^1 \text{ and } R^2 = \text{same or different H, Alk or hydroxalkyl or may form}$ part of a heterocyclic ring; W = H, Alk or alkoxy), e.g.

are fluorescent brightening agents especially applicable in combination with soap or other detergents.

Fluorescent Brightening Agents

DuP Compounds of formula-

E.S.

USP 2,700,043

(R = 2-phenylbenzothiazole or homologue thereof; X = sulphamyl or mono- or dialkylsulphamyl; Y = H, SO₂H, sulphamyl, mono- or dialkylsulphamyl; X and Y are attached to homocyclic nuclei on opposite sides of the triazole ring), e.g.

$$\begin{array}{c|c} H_{a}C & N & N & N \\ \hline & SO_{a}H & N & N \\ \hline & N & N \\ \hline & N & SO_{a}NH_{a} \\ \end{array}$$

are fluorescent brightening agents having good affinity for most fibres and particularly for nylon. When on the fibre they are unaffected by hypochlorite and other bleaching agents.

USP 2,700,044

Compounds of formula-

(R = 4'-radical of 2-phenylbenziminasole, Bz-methyl-2phenylbenziminazole or the monosulpho derivative of one of these; X = a water-solubilising acid radical; n = 1 or 2), e.g.

have similar properties to the above.

C.O.C.

Fluorescent Brightening Agents

BP 741.798 The carboxylic acid amides, urethans or ureas of 4aminonaphthalene-1:8-dicarboxylie acid imides show bluish to yellow fluorescence under ultraviolet radiation. They are used as fluorescent brightening agents for all types of fibres and many types of plastics, being especially suited for use with polyvinyl chloride polymers or copolymers. They also markedly brighten inorganic pigmenta, e.g. alumina, blane fixe or lithopone.

Blue N-Substituted Diaminoanthraquinonedicarboxylic Dyes for Acrylic Fibres USP 2,701,801

The alkyl ammonium salts of compounds of formula-

(R1 and R2 = Alk of 1-4 C; R3 = Alk of 1-4 C or aralkyl (R° and R° = Alk of I-4 C; R° = Alk of I-4 C or aralkyl (aryl is benzenoid); n=2 or 3; A= organic or inorganic ion) dye acrylic fabrics direct to yield blue dyeings which are extremely fast to light. Thus 2-(3-dimethylaminopropyl)-4:11-diaminoanthra[2:3-c]pyrrol·1:3:5:10-tetrone is dissolved in o-dichlorobenzene (300) by heating to 150°c, for 10 min. and then cooling to 60°c. Dimethyl sulphate (5) is added and the mixture stirred at 60°c. for 45 min. The mass is cooled to 25°c., filtered, and the solid product washed with anhydrous acetone and dried. It does polyagrylonitrile filters bright greenish blue dried. dried. It dyes polyacrylonitrile fibres bright greenish blue and is probably of formula—

Blue Anthraquinone Dyes for Cellulose Acetate and Terylene: Intermediates for Dyes for Acrylic Fibres DuP USP 2,701,802

Compounds of formula-

 $\begin{array}{lll} (R^1 = -CH_2CH_2-, -CH_2CH_2CH_3-, -CH_2\cdot CH(CH_3)-, \text{or} -CH_2\cdot CHOH \cdot CH_3-; & R^2 = H, & CH_3, & C_1H_4, & CH_2CH_2OH & \text{and} \\ -CH_2CH_2NH_2; & \text{whereone } R^2 = CH_2CH_2OH & \text{or} & CH_2CH_2NH_2 \end{array}$ then the other \mathbb{R}^n must $= \mathbb{H}$) are blue disperse dyes for cellulose acetate and Terylene. They have excellent fastness to burnt gas fumes and build up to deeper dyeings than do 1:4-diaminoanthraquinonedicarboximides which do not contain the above substituents on the imide N atom. They may also be used as intermediates for the dyes described in $USP\ 2,701,801$ (above). In one example 1: 4 - diamino - 2: 3 - anthraquinonedicarboximide (46), ethylenediamine (23) and Cellosolve (168) are stirred for 1 hr. at 105-110°c. and 7 hr. at 115-120°c. The mass is cooled to room temperature, filtered and the solid washed with methanol and then with water and dried. An 84% yield of-

is obtained. It dyes cellulose acetate and Terylene greenish blue of excellent fastness to light and washing. C.O.C

Benziminazolocarbocyanine Dyes Eastman Kodak Co.

Dyes of formula-

USP 2,701,198

(R1 and R2 = same or different Alk; n = 1 or 2; X1 = acid radical), e.g. 5:5':6:6'-tetrachloro-1:1':3:3'-tetrachlybenziminazolocarbocyanine iodide, used in conjunction with dyes of formula—

(R³ and R⁴ = same or different Alk or together = alkylene; X^3 = acid radical; Z^1 and Z^2 = atoms to complete a 5- or 6-membered nucleus), e.g. 1'-ethyl-3methylthia-2'-cyanine iodide, are photographic super-

Polymethin Dyes Kodak

Dyes of formula-

BP 740,770

CAO

R'N(CH:CH),C:CH-C-(CH:CH),d-CH:C(CH:CH),mNR2 C--C:A :...Q....

(R1 and R2 = same or different Alk; A = 8 or O; Q = atoms to complete a subst. or unsubst. 5- or 6-membered ring; Z^1 and Z^3 = atoms to complete the same or different, subst. or unsubst. 5- or 6-membered ring; s., d and m each = 0 or 1), e.g. 5-[di(3-ethyl-2-benzthiazoylidene)-teopropylidene]-1-methyl-2-thiobarbituric acid, are not precipitated by the usual anions. They are prepared by condensing suitable merocyanine dyes with cyclammonium quaternary salts.

Caramel of High Tinctorial Power Union Starch & Refining Co.

USP 2,701,768 Caramel colour consists of highly polymerised compounds of great tinctorial power colloidally dispersed in a true solution of sugars and variously modified sugars. On dialysis use of suitable filters results in complete separation of the dispersed colouring material from the dissolved sugar and sugar derivatives.

Rutile-containing Composite Pigments

National Lead Co An alkaline earth metal slurry is boiled with a titanium sulphate solution, the solution containing 5-50% of the Ti needed to form the composite pigment. The remainder of the Ti is then added as titanium sulphate solution and the whole boiled to complete precipitation. The precipitate is filtered, washed and calcined at 900-1000°c.

American Cyanamid Co.

If the manufacture of ultramarine is carried out in a gastight electric resistance furnace then the excess sulphur can be wholly recovered in elemental form, a 24 hr. cycle is readily achieved, the advantages of the briquetted mix and SO₂ oxidation of primary ultramarine are fully retained and the equipment is small, compact and cheap compared with the Beardsley and Krupps tunnel kiln. C.O.C.

Dewatering of Aqueous Pastes or Slurries of Finely Divided Hydrophobic Solids Jones Gas Process Co. BP 741,135

Maximum dewatering is obtained of e.g. slurries of carbon black, by mixing them, if necessary with addition of further water, with a controlled amount of a volatile water-immiscible liquid which has a higher adhesion tension than water for the solid. The separated water is removed and the solid dried by evaporating the other liquid and the small amount of water left in the solid.

Surface-active Compositions containing 4-Methyl-7diethylaminocoumarin as a Fluorescent Brightening Agent (III p. 60)

Fluorescent Brightening Agents in Detergents and Washing Processes (VII p. 70)

Spin Dyeing of Superpolyamides (VIII p. 71) 1:3:5-Triazines and their Separation by Paper Chromatography (XIV p. 76)

V-PAINTS; ENAMELS; INKS

Effect of the Electrolyte Content of Inorganic Pigments on the Swelling of Films of Polyvinyl Acetate when exposed to Water K. Meier and G. Schulz

Farbe und Lack, 61, 220-225 (1955) PATENTS

Rust-inhibiting Aluminium Paint containing Strontium Chromate

Reynolds Metals Co. USP 2,701,772 Strontium chromate is ground in mineral spirits, coumarone resin solution, boiled oil, or the like and then mixed with aluminium flake. One part of strontium chromate to 1-4 parts of aluminium is used. The aluminium flake retains its leafing properties.

Paints, Lacquers, Printing Inks, etc. BP 740,776 ociété Française Duco

In making lacquer, etc. containing resinous binders use of a dispersing agent can be dispensed with if instead there is used some of the resinous binders in presence of a fairly large proportion of solvent for the binders. On vigorous agitation rapid wetting and dispersion of the pigment is obtained. The resulting dispersion can be diluted by adding the rest of the components to yield a homogeneous product without interfering in any way with the dispersion of the pigment.

Printing Ink Varnish J. M. Huber Corpn.

BP 741,750-1 Resin salts formed from a water-insoluble resin of V. <90 and a base of dissociation constant $>1\times10^{-6}$ at 25°c, are dissolved in a 20-60% aqueous solution of urea or a mixture of 1 part by wt. of such a solution with 2.3 parts by wt. of glycerol, a monohydric alcohol, ether or ester of a glycol (glycol of 2–8 C and containing only C, H and O), are cheap varnishes for printing inks which can be set very quickly by a variety of methods. C.O.C.

Cellulose Nitrate Lacquers

BP 741,521 Midland Silicones Lacquers containing 25-90% by wt. of cellulose nitrate and 10-75% of a silicoalkyd resin yield coatings of exceptional resistance to weathering. C.O.C.

Flushing Process for Pigments (IV p. 64)

VI-FIBRES; YARNS; FABRICS

Use of Radioactive Tracers in Textiles C. E. Crompton

Textil Praxis, 10, 971-979 (Oct. 1955) A review of the possible outlets for radioactive isotopes in the textile industry, including the suggested use of tracers in controlling the contamination of printing pastes in multicoloured printing, movement of fibres, and control of scouring efficiency.

B.K. control of scouring efficiency.

Two Recently Observed Forms of Textile Damage O. Oldenroth

Melliand Textilber., 36, 1168-1170 (Nov. 1955)

"Reactivity" of Celluloses used in Viscose Manufacture

F. I. Korchemkin and L. P. Zherebov

J. Appl. Chem. U.S.S.R., 28, 872-876 (Aug. 1955) The filterability of viscose prepared under standard conditions from various wood and cotton cellulosessuitable for viscose preparation, and some not—is taken as a measure of the "reactivity" of the cellulose used. The reactivity is correlated with the behaviour of the fibre in Schweizer's reagent, the tendency for the fibre to form bead-like swellings being especially associated with lack of reactivity. It is concluded that the weakening and breakdown of the outer layers of the walls of the fibres in the process of the preparation of cellulose for xanthation, together with other changes in the fibres that result from these changes in the outer layers, are the main factors determining the reactivity of the cellulose. A.E.S.

Preparation of Denitrated Cellulose

Martin Vicente

XXVIII Congrès International de Chimie Industrielle Chim. et Ind., 74, 167 (Oct. 1955)

Membranes of cellulose are prepared by denitration of cellulose nitrate films.

Solvation and Swelling of Cellulose Fibres-Action of Sodium Hydroxide J. Chedin

XXVIII Congrès International de Chimie Industrielle Chim. et Ind., 74, 170 (Oct. 1955)

Results are given on the fixation of caustic sods on cellulose macromolecules and on the accessibility of native fibres to increasing concentration of caustic soda. A theory is outlined based on the existence of hydrates Na⁺OH⁻,zH₂O in solution, which exchange 3H₃O with 3OH groups in the cellulose macromolecules. Mcroerination phenomena constitute the steric aspect of this formation of mixed hydrates.

Swelling and Absorption by Cellulose Acetate
W. R. Moore and J. Russell
J. Polymer Sci., 18, 63–86 (Sept. 1955)
Preferential absorption by, and swelling of, cellulose acetate fibres and films in binary solvent-type liquid-hexane mixtures are studied at 25°c. Factors affecting absorption of solvent-type liquids by fibrous secondary acetate include the activity of the liquid and its chemical type, the swelling power of the mixture, and steric effects. Preferential absorption of ketones and most esters seems to be independent of acetyl content and structure of polymer. Absorption of methyl formate, acetic acid, and heterocyclic bases by the triacetate is less than by the secondary acetate. At higher concentrations cresols and chlorinated hydrocarbons are absorbed to a greater extent by the primary acetate. Such variations in absorption are ascribed to steric effects and to elimination of hydroxyl groups on complete esterification. At higher concentrations of solvent-type liquid some hexane seems to be absorbed. There is no general relationship between swelling and the Hildebrand solubility parameter δ of the solvent-type liquid, but δ values for the mixtures just causing complete solution of the acetates are not very different when the mixtures contain solvents of similar chemical type.

Cystine-Cysteine and Total Sulphur Contents in Wools from Different Pastures and Breeds in Spain M. D. Astudillo, A. S. Belda, M. I. Alonso, and A. Valdecantos

XXVIII Congrès International de Chimie Industrielle

Chim. et Ind., 74, 169 (Oct. 1955)

The cystine-cysteine (I) content was determined
colorimetrically (Sullivan-Hess-Howard method) and the
total sulphur (II) estimated by the semimicro peroxide
bomb (Parr) method, giving limits (I) 11-6-13-9% and (II)
3-6-3-8%. The values are discussed in relation to values 3.6-3.8%. The values are discussed in relation to values determined elsewhere.

Composition of Unsaponifiable Matter in Spanish Wool Greases

F. Martín Panizo and J. L. García Bilbao

XXVIII Congrès International de Chimie Industrielle Chim. et Ind., 74, 182 (Oct. 1955) A preliminary study indicated 49–51% unsaponifiable.

The "steroid fraction" was later examined, and was considered to be of greater interest than the fractions obtained by the Johnson methanol method. The efficiency of fractionation of the latter method in the separation of sterols is discussed, and the composition of the unsaponifiables of Spanish wool greases is given, the possi-bility being examined as to whether the components are normal or result from recovery, storage, or subsequent processing. 8.R.C.

Absorption and Desorption of Hydrobromic Acid in Wool B. Olofsson

Trans. Faraday Soc., 51, 1583-1591 (Nov. 1955) Calculations of diffusion constants for sorption of HBr in wool are made. Absorption and desorption coefficients are effectively of the same magnitude. They decrease with time for the first minute and then attain a fairly constant value. Calculated curves for distribution of the sorbed substance in the fibre at different times are given. The magnitude of the diffusion constants is accounted for by the surface charge of the fibres. Activation energies are found to be the same for absorption and desorption. Their magnitude indicates that some work is necessary for penetration of the fibre phase in acid diffusion.

Formation and Stability of the two Modifications of

Mk. Chem., 86, 379-384 (June 1955)

Spun silk fibres consist of Silk II, which on recrystallisation always gives X-ray diagrams of II. That part which is not decomposed is still II even after heating at 200°C. in nitrogen for 10 hr. Silk II must therefore be the stable modification. Silk I, obtained by drying native silk gel, after heating gives an X-ray diagram corresponding to I+II. It had been shown previously that mechanical action converted I into II. However, quick evaporation of solutions of "renatured" silk yields I, slow evaporation II. H.E.N.

Fibrillar Structure of Fibres. IV-Fibrillar Structure of New Synthetic Fibres K. Miyasaka and Y. Watanabe

J. Soc. Textile Cellulose Ind. Japan,

11, 465-468 (Aug. 1955) In studying mechanical action on synthetic fibres in a swollen state with an optical microscope, it was observed that all the types of new synthetic fibres used in this experi-ment show distinct fibrillation in accordance with the crystallinity. In the initial stage of disintegration obtained by application of slight mechanical action, the larger fibrils were revealed, and these appeared to be oriented parallel to the fibre axis. The external appearance assumed after a given amount of mechanical action is peculiar to each type of fibre. It seems probable that all textile fibres exhibit fibrillation when they are mechanically disintegrated in a moderately swollen state. There is no direct evidence that fibrils exist in the synthetic fibres before breakdown treatments.

1,n-Polyamides and Methylenebisamides

Cannepin
XXVIII Congrès International de Chimie Industrielle Chim. et Ind., 74, 169 (Oct. 1955) Since 1,2-polyamides differ from the remainder of the group in e.g. higher m.p. and strong crystallinity, the aliphatic methylenebisamides have been studied.

S.R.C.

Preparation of Polyvinyl Chloride Fibres T. Yoshioka

-Heat Treatment of the Fibre with Water, Boric Acid, and Formalin J. Soc. Textile Cellulose Ind. Japan,

11, 449-454 (Aug. 1955) Variations of denier, tensile strength, elongation, and heat shrinkage caused by heat treatments with water, 10% boric acid, and 35.7% neutral formalin at 60-100°c. for 20-390 min. were studied. Denier, tensile strength, and wet : dry tensile strength ratio show almost no change compared with untreated original fibre, but elongation is decreased nearly to 20%, and wet : dry elongation ratio is increased to about 100%. Heat shrinkage is decreased by treating at higher temperature and for longer time. In these treatments curves of heat shrinkage changed to a different shape from that of the untreated original fibre.

VI-Treatment of the Fibre with Thermosetting

The influence on the tensile strength, elongation, and heat shrinkage of polyvinyl chloride fibres of treatment with 5 or 10% thermosetting resin solutions, such as urea-formaldehyde and melamine-formaldehyde, studied. Denier and tensile strength are increased by dipping in resin solution and curing at 130°c. for 30 min., and elongation is decreased, but fixed resin on the fibre is lost by soaping for 30 min., so that these effects are not

shown, and the inhibitory effect on heat shrinkage is not large compared with heat treatment. However, after treatment with a coagulating bath which contains resin, the fixed resin remains in the fibre. Consequently the inhibitory effect on heat shrinkage is larger than with

Polyvinyl Chloride

XVII— Cross-sectional Shapes of Pure Polyvinyl

Chloride Fibre

T. Imoto and K. Inubushi

J. Soc. Textile Cellulose Ind. Japan,

11, 369-375 (July 1955) Various cross-sectional shapes were observed with pure polyvinyl chloride fibres spun from four types of solvent-coagulating agent systems under several different conditions. Thus, four series of shapes obtained from tetrations. Thus, four series of suspess covariant to the hydrofuran-water, nitrobenzene-methanol, mesityl oxidemethanol, and carbon disulphide-methanol systems were uncertain-elliptical-dumbbell, flat dumbbell, uncertain-circular-elliptical, and uncertain-circular types respectively, with their degree of coagulation. Also, these shapes differed from each other according to the winding velocity and/or bath temperature. These varieties of cross-sectional shapes could be accounted for with some data derived from model experiments such as coagulation on a microscope cover glass and osmosis between solvent and congulating agent.

XVIII- Thermal Shrinkage of Pure Polyvinyl Chloride Fibre

T. Imoto

Ibid., 375-379 (July)

XIX-Thermal Decomposition of Pure Polyvinyl Chloride Fibre T. Imoto

Ibid., 379-384 (July) The following points were confirmed for the thermal dehydrochlorination of pure polyvinyl chloride fibre spun from tetrahydrofuran (THF) solution in a stream of N_2 —(1) Almost 1% of the solvent remains for a long period in the fibre. (2) On thermal decomposition of the fibre HCl is liberated, and also probably tetramethylene chlorohydrin. (3) Rates of decomposition of fibres drawn to twice and four times their lengths are about 88% and 73%, respectively, of the rate for the undrawn fibre.
(4) Likewise, the decomposition of the fibre drawn to higher ratio showed higher activation energy.

XXIII—Some Chemical Treatments to prevent Thermal Shrinkage of Pure Polyvinyl Chloride Fibre

T. Imoto

Ibid., 440-445 (Aug.) Treatments with NH₃, NaNH₃, formalin, etc. were applied and compared on pure polyvinyl chloride spun from tetrahydrofuran solution and drawn to several ratios. These treatments were effective for undrawn fibre or fibre drawn to a small ratio, but had no effect on highly drawn fibre. The effectiveness of these treatments varied in following order: (formalin + NaOH) > (formalin +HCl) > (ammonia + ZnCl₂) > formalin only > sodamide > ammonia only > steam > aq. NaOH > heat only.

XXVII- Thermal Shrinkage of Pure Polyvinyl Chloride Fibre (2)

T. Imoto

Ibid., 445-449 (Aug.) Shrinking velocities of pure polyvinyl chloride fibres, spun from tetrahydrofuran solution and drawn to various ratios, were measured by photography with a camera on a small time-scale, since the degradation period of thermal shrinkage of these fibres is so short. The shrinking behaviour consists of two parts, one above and one below the temperature of the second-order transition point.

Polyvinyl Chloride Fibres of Different Degree of Polymerisation and Composition N. Mori, T. Tanaka, and K. Fukumi J. Soc. Textile Cellulose Ind. Japan, 11, 530-533 (Sept. 1955) Pure polyvinyl chloride fibres with different D.P. have

similar mechanical behaviour, but the shrinkage in hot water is affected by the D.P. Except for shrinkage in hot

water, there is also no notable difference between fibres from pure polyvinyl chloride and those from a copolymer which contains about 5% vinyl acetate. The effect of heat treatment is greater with fibres from polyvinyl chloride with higher D.P. and with those from pure polymers than with those from a copolymer.

Fibres from Vinylidene Chloride Mixed Polymers H. Anders

Textil Praxis, 10, 1006 (Oct. 1955) Physical and chemical properties of fibres from vinylidene chloride mixed polymers with particular reference to their resistance to a wide range of inorganic and organic chemicals.

Preparation of Synthetic Fibres from a Mixture of Polyvinyl Alcohol and Polyvinyl Cyanoethyl Ether M. Negishi, I. Sekiguchi, N. Mori, and S. Okada J. Soc. Textile Cellulose Ind. Japan,

11, 384-388 (July 1955)

Easily dyeable fibres can be obtained from a solution of a mixture of polyvinyl alcohol and polyvinyl cyanoethyl ether whose degree of cyanoethylation is lower than 25 mol. %. The mechanical properties of these fibres are similar to those of ordinary vinylon.

AUTHORS

Plasticised Cellulose Esters and Ethers resistant to Light and Heat

BrC Cellulose acetate or other cellulose ester or ether when plasticised with trichloroethyl phosphate or other halogenated plasticiser is protected against liberation of HCl when heated and/or exposed to light if an alkali metal salt of an aromatic carboxylic acid, e.g. 1% (on the wt. of plasticiser) disodium phthalate, is added to the plasticiser. Cellulose acetate containing such plasticisers can be dved with aminoanthraquinone dves to yield dyeings which are of very good fastness to light and/or heat.

Cellulose Acetate which imparts Resistance to Gas Fume Fading to Dyes applied to it

BP 740,649 Cellulose acetate containing 1-5% of a polymer containing carboxyl groups or groups which are converted into carboxyl groups by water or aqueous alkalis, e.g. equimolecular condensate of styrene and maleic anhydride. when coloured with dyes which are not normally resistant to gas fume fading yields dyeings or prints of excellent resistance to gas fume fading.

C.O.C.

Solutions of a-Chloroacrylonitrile Polymers BP 741,236

Solutions of a-chloroacrylonitrile polymers in tetrahydrofuran, its homologues or ketonic solvents are stabilised by presence of small quantities of HCl, thus enabling them to be used e.g. for the production of fibres. BP 741.237

Solutions of a-chloroacrylonitrile polymers in nitromethane are stable at temperatures up to 60°c

BP 741,238 The solubility of a-chloroacrylonitrile nitromethane is considerably increased by addition of a phenol or a nuclear homologue thereof and/or of water.

C.O.C.

Dyeable Fibre-forming Acrylonitrile Copolymer Allied Chemical & Dye Corporation

A heterogeneous mixture of acrylonitrile-methyl methacrylate copolymer molecules (made so that those formed in the early stages of polymerisation are much richer in methacrylate than those formed in the later stages) yields filaments which can be stretched to 10 times or more their original length, have excellent resistance to acids, alkalis and shrinkage in boiling water and good C.O.C. affinity for disperse dyes.

Increasing the Affinity of Acrylonitrile Polymers for Acid Dyes

USP 2,701,244 Chemstrand Corpn. Treating a dissolved acrylonitrile polymer with compounds of formula-



 $(R^1 = Alk \text{ of } < 11 \text{ C}; R^2 = H \text{ or } Alk \text{ of } < 4 \text{ C}), e.g.$ methyl isonicotinate, in presence of an alkali-metal alkoxide of <5 C, e.g. sodium methoxide, results in fibres formed from the treated polymer having very good affinity for acid dyes.

Fabrics having a Wool-like Finish

Fabric composed of a blend of a non-shrinkable fibre with one that shrinks on wetting, e.g. highly stretched viscose staple fibre blended with fine-denier normal viscose staple fibre, when wetted yields a bulky having the warmth characteristic of wool.

Research on the Crease Resistance of Cloth. V -- Effect of the Yarn Density on the Crease Resistance of Fabric (X p. 73)

The Concept of Swelling Anisotropy, III—Swelling Anisotropy and Elongation on Wetting—Applica-tions (XIV p. 76) III- Swelling

VII— DESIZING; SCOURING; CARBONISING; BLEACHING

Wool-scouring Procedures

H. C. Borghetty

Amer. Dyestuff Rep., 44, P 726-P 729 (10 Oct. 1955) Mechanical aspects of scouring are briefly surveyed, with appears on raw wool scouring.

J.W.B. emphasis on raw wool scouring.

Fluorescent Brightening Agents in Detergents and Washing Processes L. Maur

XXVIII Congrès International de Chimie Industrielle

Chim. et Ind., 74, 184 (Oct. 1955) Specific properties of fluorescent brightening agents are considered, and conditions for their use in washing, washing powders, brightening of soap and detergents, in rinsing, and in dry cleaning are enumerated.

Continuous Cotton Bleaching with Silicate-free Peroxide Solutions

P. Feldmann and B. K. Easton

Amer. Dyestuff Rep., 44, P 745-P 748 (24 Oct. 1955)
Although an outstanding peroxide stabiliser, sodium silicate has fallen into disrepute in continuous bleaching ranges because of scale formation and cloth staining. However, it appears that Ca and Mg silicates, even in small quantity, serve an essential function in the stabilisation of H₂O₂. Experiments are described in which the decomposition of H₂O₃ liquor in presence of phosphate and a small amount of added CaCl₃ is studied, and recipes are given whereby effective controlled bleaching can be carried out J.W.B. with these additives.

PATENT Bleaching Low-grade Cotton Chicopee Manufacturing Co.

Modification of BP 702,300 (J.S.D.C., 70, 134 (1954)). Low-grade cotton, either loose or in fabrics, is steeped in an inactive solution of a per compound for at least 90 min. at 15°c. to 75 min. (the time diminishing with increase of 15°C. to 75 rain, the table differentiating with the temperature) at 50°C., or for temperatures between 50–55°C. for 75–90 min. (the time increasing with increase of temperature). The solution is then activated and the material kept in it until the per compound is exhausted. CO.C.

Use of Radioactive Tracers in Textiles (VI p. 68)

VIII— DYEING

Current Uses of Vegetable Dye Extracts in the Textile Industry

G. Parsy

Teintex, 20, 859-877 (Nov.), 951-972 (Dec. 1955)

Logwood extract is used for the production of blacks on felts, vegetable fibres, wood, leather, furs, silk, nylon, and acetate rayon and in printing. The haematoxylin may be partly or fully oxidised to haematin and

sold in liquid, paste, or crystallised form. The tinctorial value of such extracts can be assessed by means of laboratory dyeings with extracts exidised to various degrees, e.g. with NaNO₂, or colorimetrically by means of the violet complex given by haematin with 8nCl₂ in HCl. 8.R.C.

Cutting, Dyeing, and Finishing of Velveteen and G. Capron

Teintex, 20, 886-910 (Nov. 1955) Historical and geographical introduction. The number Historical and geographical introduction. The number and width of the races distinguish the various types of fabric. The back is raised prior to cutting, which may be carried out with straight knives on Keighley and Netherwood machinery or by circular machines. Desizing and boiling follow. After brushing, singeing, and bleaching the fabrics are ready for dyeing on jigs. A number of sulphur, direct, basic, solubilised vat, and vat recipes are given. Under finishing, back-sizing, softening, waxlustring, and waterproofing are described.

8.R.C.

Donnan Membrane Equilibrium and Direct Dyeing of Regenerated Viscose Sheet—III K. Nishida

J. Sec. Textile Cellulose Ind. Japan,
10, 303-305 (1954)
Chem. Abs., 49, 15243 (10 Nov. 1955)
The amount of dye absorbed by regenerated cellulose sheet from a bath containing 0-05-0-4 g. of Diamine Blue
2B and 1-40 g. of NaCl per litre agreed approximately with the value calculated from the Neale equation derived from the Donnan membrane equilibrium at 60°c. but not at

Union Dyeing of Acetate-Viscose Rayon Blended Fabrics. V— Union Dyeing Y. Matsuda and T. Adachi

J. Sec. Textile Cellulose Ind. Japan, 11, 408-413 (July 1955) On the basis of the C.I.E. diagrams obtained from the previous papers, 50: 50 acctate-viscose rayon blended fabrics were dyed in solid colours with acctate and direct cotton dyes by the sc-called "single-bath method". In this paper the combination of x-y coordinates and brightness Y of the given dyed toxtile was first found, then the required ratio of the dyes was determined by means of the C.I.E. diagram, and fabrics were dyed. In most cases slight colour differences were observed between the pattern and the dyeings. So the proportions of the dyes were amended, and fabrics were dyed again. Thus, trials were carried on until results very close to the original were obtained. Six colours were tried in the same way, but this method may be utilized over the wider range of the diagram.

Spin Dyeing of Superpolyamides L. LeThierry d'Ennequin

Teintex, 20, 879-885 (Nov. 1955)
Compounds containing no solubilising groups may be mixed as powders with flakes of superpolyamides, and the mixture fused at 220-300°c, in nitrogen for melt-spinning. If solubilising groups are present (e.g. -80₂Na), the flakes may be coloured in an aqueous solution and then spun. A list is given of compounds obtained by condensation of di- or tetra-basic acids with di- or tetra-amines and their colours when applied to Rilsan-

Naphthalenetetracarboxylic acid 1:8-Naphthylenediamine

Blue-violet

Some members of this list figure in the range of Aminoid (Fran) colours for the spin dyeing of superpolyamides.

Producing Very Fast Dyeings or Prints with Acid Dyes on Organic Derivatives of Cellulose BASF

Very fest dyeings or prints are obtained with water-soluble acid dyes containing one sulphonic acid and/or one sulphamide group on cellulose acetate or other organic derivative of cellulose by treating them before or during dyeing and printing with a compound of formula-

 $(X = CH_2, CO \text{ or } NH; R^3 \text{ and } R^3 = H, Alk, aminoalkyl)$ or oyanoalkyl). Thus acetate rayon fabric (100 parts) is treated in water (2000) containing the formaldehyde bisulphite compound of the compound-

at 75-80°c. for 30 min. It is then rinsed and treated in water (2000) containing 1-amino-4-m-phenylamino-anthraquinone-2-sulphonic acid (2) and commercial H₂8O₄ (2) at 75-80°c. for 30 min. The deep blue dyeing so obtained is very fast to light and wet proces

Dyeing Polyacrylonitrile with the aid of a Copper Salt and Furfural

DuP Strong level dyeings are obtained by dyeing polyacrylonitrile with acid dyes in an aqueous bath at <222°r, and at pH 1.5-5.0 in presence of 0.5-2.5% (on the wt. of the fibro) of cuprous ion and 1-10% of furfural.

C.O.C.

Rack for Dyeing Stockings (I p. 59)
Nature of the Internal Hydrogen Bond (IV p. 60) Synthesis of some Aromatic Amines containing Tri-fluoromethyl Groups and their Use in Azoic Dyeing

Selectivity Factor-Ita Influence on Padding (X p. 72)

IX-PRINTING

Behaviour in Printing and Physical Properties of Alginate Thickenings K. Schmidt

Melliand Textilber., 36, 1144-1146 (Nov. 1955)
The behaviour of three types of alginate as regards sharpness of printing and viscosity-fluidity characteristics in soft water, hard water ± Calgon, acetic acid, soda, and potash is discussed and illustrated.

8.R.C.

Direct Printing of Vat Dyes by the Rongalite C-Potash Process-I G. Bernardy

Melliand Textilber., 36, 1151-1159 (Nov. 1955)
The types of sodium formaldehyde-sulphoxylste, alkali, auxiliaries, and specialised chemicals, and thickenings suitable for the process are described. Recipes for various s are quoted, and reference is made to the storage and durability of the prints before steaming.

Printing of Polyacrylonitrile Fabrics R. Walter

Melliand Textilber., 36, 1146-1151, 1174-1175

(Nov. 1955)

Direct printing with disperse, triphenylmethane, polymethin, vat, solubilised vat, acid, and pigment dyes is discussed. Processes based on the discharging of padded triphenylmethane or polymethin dyes, disperse dyes, acid dyes, and solubilised vat dyes are outlined. S.R.C.

PATENTS

Maintaining Register in Cylinder Printing Machines Cranston Print Works Co. BP 741,896 A method of obtaining correct warp and weft ways registration easily and quickly. Once obtained, registra-tion is maintained for long periods by maintaining uniform tension in the material passing through the machine.

Printing Screens

R. W. Henningsgard USP 2,700,930 The design to be reproduced is applied directly to a

transfer film. On the transfer being applied to the gauze of a printing screen, only the parts of the film not covered by the design are transferred to the gause. This is a quick and cheap method of making printing screens. C.O.C.

Relief Images suitable for Direct Use as Printing Plates DuP BP 741.294

Printing T. N. Gaunt and N. V. Watson

A print made with a comparatively sticky ink is dusted with a resinous powder before the ink is dry. Surplus powder is removed and the print treated with a liquid which causes the powder to agglomerate and become bonded to the ink.

Electrostatic Printing

BP 741,259 Huebner The ink on the block or the like and the material to be printed are each charged with unidirectional electrostatic charges of opposite polarities and are then brought near to each other in a pulsating field of force. The charges of the field of force co-operate with the charges on the liquid

and material to transfer the former to the latter.

Transfer Paper

McLaurin-Jones Co. USP 2,700,619 The backing paper for transfers is coated with a size

containing dimethyl hydantoin formaldehyde, the transfer being then printed on the coated surface. On moistening the paper it is readily detached from the printed coating.

Transfers and Apparatus for making and applying

American Photofoil Corpn. USP 2,700,629

Transfers suitable for use on hard surfaces, e.g. glass, or on a ft thermoplastic surfaces or textiles sized with soft thermoplast materials consist of a flexible unstretchable backing and a pigment or dye dispersed in a matrix resin which is not compatible with the backing but is highly compatible with the surface to be decorated. The matrix resin contains a flow agent which has a lower mol. wt. and a lower temperature of flow than the resin and is more readily affected by solvents. Apparatus for making and applying the transfers is described.

Transfers for marking Extensible Objects

BP 741,190 London Rubber Co. A transfer is coated with an aqueous dispersion of rubber containing a plasticiser. On drying this leaves a tacky film on the transfer. This film is stable for long periods and has strong adhesive properties when applied to rubber so that on being transferred the marking is strong and permanent. The transferred film is very

C.O.C. Electro-optical Production of a Black Printer for Use in making Four-colour Reproductions

Time A method of improving colour prints by modifying the high-light and shadow densities in a black printer.

Use of Radioactive Tracers in Textiles (VI p. 68) Current Uses of Vegetable Dye Extracts in the Textile

Industry (VIII p. 70)
Producing Very Fast Dyeings or Prints with Acid Dyes on Organic Derivatives of Cellulose (VIII p. 71) Coated Paper for making Planographic Printing Plates (XI p. 75)

X-SIZING AND FINISHING

Selectivity Factor-Its Influence on Padding

Amer. Dyestuff Rep., 44, P 733-P 736 (10 Oct. 1955) Selectivity factor is the ratio of the amount of finishing agent which is actually deposited on fabric by padding to the amount that would be deposited were there no preferential adsorption of reagent or water. Experiments are carried out on desized, scoured, and peroxide-bleached

cotton sheeting, using starches, thermoplastic and thermo-setting resins, elastomers, and repellents. Starches show a dectivity varying with viscosity, high-viscosity starches showing preferential water adsorption, whereas those of low viscosity are themselves preferentially adsorbed. With thermosetting resins the relation is complex, but there is in general water selectivity, though improved resin pick-up is promoted by increasing the nip pressure. Thermoplastic resins are not so much affected, but the factor increases with increasing viscosity, which is of interest because such resins are often used as substitutes for starch, which behaves in the opposite way. The two elastomers (butadiene-acrylonitrile copolymers) did not behave exactly alike, though a strong solid selectivity was found; a silicone emulsion repellent behaved similarly. It appears in general that surface deposition agents have a solid selectivity, and interior reactants a water selectivity.

Effect of Light on Metallised Fabrics G. Heyl and M. Kramer

Melliand Textilber., 36, 1161-1166 (Nov. 1955)
The application of metal, e.g. Al bronze, to textiles increases the photochemical resistance of the fibre, protects it from heat rays, and increases the heat insulation of clothing. Methods of measuring these effects in textiles treated with metals in various ways are discussed, and uses of the fabrics, e.g. heat-protective clothing, curtains, car hoods, and balloons, are reviewed. S.R.C.

Effect of Detergents on the Fluorescence Fluorescent Brightening Agents A. Yabe and M. Hayashi

J Chem. Soc. Japan, Ind. Chem. Sect., 58, 345-352 (1955) Chem. Abs., 49, 14328 (25 Oct. 1955)

After repeated washing tests cotton treated with Kaycoll BB (a triazylstilbene fluorescent brightening agent) showed increased fluorescence. This effect (called "sensitising") was attributed to the detergent present when the Kaycoll was applied. Anionic surface-active agents have a bigger effect than non-ionic or cationic

Silicone Finishes for Wool

R. Antoine

XXVIII Congrès International de Chimie Industrielle Chim. et Ind., 74, 170 (Oct. 1955)

The range of available finishes is completed by two emulsions, one giving an improvement in handle and stain resistance, and the other an increase in resistance to felting, whilst conferring most of the other properties of a silicone finish.

Resin Finishing of Silk Fabrics. II-Relation between Resin Finishing and Crease Resistance of Silk Habutae

A. Kuwahara

J. Soc. Textile Cellulose Ind. Japan

11, 405-408 (July 1955) The following six finishes were studied—(i) gelatin, polyvinyl acetate, (iii) polymethyl acrylate, (iv) yvinylidene chloride, (v) polymethyl acrylate-vinyl polyvinylidene chloride, (v) polymethyl acrylate-vinyl acetate copolymer, and (vi) methylolmelamine. It was found that (vi) and (v) were more adhesive than the others. For reversible angle of crease, (i), (ii), and (vi) are preferred to the others. For long-period creasing, however, (iv), (v), and (iii) are preferred. AUTHOR

Flame Resistance of Military Textiles

A. J. McQuade

Amer. Dyestuff Rep., 44, P 749-P 751 (24 Oct. 1955) The use in fireproofing of triallyl phosphate, its bromo-form adduct polymer and chloroform copolymer, and tetrakishydroxymethylphosphonium chloride resin are briefly discussed, and some graphs are shown illustrating a skin burn test (on animals) for assessing protective efficiency.

Urea-Formaldehyde Condensation

XIII- Urones

G. Zigeuner, R. Pitter, and K. Voglar

Mh. Chem., 86, 517-523 (June 1955)

XIV-N-Methoxymethyl-N'-methylurone

G. Zigeuner and R. Pitter Ibid., 524-527

2:5-Bisethyleneiminoquinol

A. Marxer

Helv. Chim. Acta, 38, 1473-1489 (Oct. 1955)

Crease-resist Finishes J. C. Sánchez

Ingeneria Textil, 22, 427-434 (Sept.-Oct. 1955) A review, including consideration of the influences of (a) the fibre (molecular and external structures and agents modifying these); (b) the yarns, weave, dyeing and finishing treatments. 8.R.C.

Crease-resistant Finish with Urea-Formaldehyde Resin. VI—Effects of the Catalysts on Crease Resistance, Strength, and Elongation of the Fabrics

J. Soc. Textile Cellulose Ind. Japan.

11, 550-555 (Sept. 1955) The relation between pH of uncondensed urea-formaldehyde solution for the crease-resistant finish or the resin content of the treated fabric and its crease resistance, tensile strength, and elongation, and the effects of the catalysts (hydrochloric acid, buffer, basic aluminium chloride, dimethylaniline hydrochloride, pyridine hydrochloride, picoline-betaine hydrochloride) on the above relations, were studied, with the following results: (1) With a given catalyst, crease resistance increases with increase in resin content of the treated fabric and decrease in the pH of the solution for the treatment. (2) At a given pH or resin content, the latent catalyst had the better effect on crease resistance. Especially, picoline-betaine hydrochloride catalyst (Nippon Patent 201,708 (1953)) shows the best effect on this property. (3) The fabric acquired the maximum increase in strength on treatment with a resin solution having a pH value of 4.4. This maximum increase of strength has the same value for each catalyst, and bears no relation to the type of catalyst. (4) The resin content of the treated fabric which gives the maximum increase of strength is not the same for all the catalysts, and the latent catalyst had its maximum point at the higher resin contents of the fabrics. (5) Elongation of the treated fabric is related to the resin content of the fabric, and not to the type of catalyst. (6) Imbibition value is also related to the resin content of the fabric, and the formation of linkages between cellulose molecular chains by the resin treatment cannot be deduced from the decrease in imbibition value. AUTHOR

Research on the Crease Resistance of Cloth 8. Ikeda and 8. Okajima

V-Effect of the Yarn Density on the Crease Resistance of Fabric

J. Soc. Textile Cellulose Ind. Japan

11, 397-405 (July 1955) Fabrics of eight structures, including plain, twill, sateen, and basket weaves, were made from the identical spun yarn, but the yarn density was varied. Half of each fabric sample was treated with a urea-formaldehyde resin. The crease resistance of the samples, treated and untreated, was measured, as described in the preceding papers in the series. In general, the crease resistance decreased and approached a constant value with increase in yarn density, but in some cases it increased after passing through a rainimum. Among the samples, the crease resistances of the sateen and the basket weaves were excellent, but they could be improved slightly by the resin treatment, while the plain, which was improved remarkably, had a lower resistance before treatment than those of the twill, sateen, and basket. Yarn density changes often during the treatment, so the evaluation of the effect of the resin finish must be made with care.

VI-Evaluation of the Effect of the Creaseresistant Finish

Ibid., 546-550 (Sept.) Crease-resistance of fabric is affected considerably by the density of the warp and weft threads as reported in the preceding paper, and this density increases generally during resin-finishing. So the effect of the finishing must be appreciated by taking this fact into consideration. After comparing many expressions for the crease-resistance of a fabric the authors propose a new method of expression.

Resin Finishing of Cellulosic Fabrics. VII-Effects of the Formaldehyde Concentration in the Treating Bath and of the Addition of Non-ionic Surface-active Agents on the Mechanical Properties of Formalised Yarns and Fabrics

S. Matsuzaki and T. Yanase

J. Soc. Textile Cellulose Ind. Japan,

11, 556-560 (Sept. 1955)

The mechanical properties of viscose rayon filament yarns and spun staple fabrics formalised under hydro-chloric acid catalyst were studied when the concentrations of formaldehyde and catalyst in the treating baths were varied and also various non-ionic surface-active agents were added. Viscose filament yarns and spun rayon fabrics were immersed in each treating bath, squeezed, and dry-heated for 10 min. at 120°c.

The concentration of formaldehyde in the treating baths was varied from 3 to 26 g./100 c.c., and to each bath was added HCl corresponding to 5% by weight of the formaldehyde contents. The weight after treatment increased in proportion to the increase in concentration of formaldehyde in the treating baths, but the effective reaction rate of formaldehyde in each case was nearly constant. The crease resistance of the fabrics was markedly improved by treatment, and the tensile strength and elongation were lowered as the formaldehyde content increased. However, on the treated filament yarns, their dry strength was lowered and their wet strength, crease resistance, and elastic recovery were all increased.

Six non-ionic surface-active agents were tested—lauryl polyglycol ether $C_{12}H_{25}$ ·O· $[C_2H_4\cdot O]_{g-1}$ ·C· $_2H_4$ OH (x=6,9,20), oleyl polyglycol ether $C_{16}H_{35}$ ·O· $[C_2H_4\cdot O]_{g-1}$ ·C· $_2H_4$ OH (x=10,15), and octadecyl polyglycol ether $C_{16}H_{37}$ ·O· $[C_2H_4\cdot O]_{15}$ ·C· $_2H_4$ OH. The concentrations were 0·3, 0·6, and 1·0 g./100 c.c. in the treating baths, which contained 12 g. H·CHO and 0·1 g. HCl per 100 c.c.

In the treated spun rayon fabrics, the addition of non-In the treated spun rayon rather, in the treated spun rayon rather including the control of the the softening, lubricant, and resilient handle. was not related to the type of hydrocarbon chain or the mol. wt. of the agents. Such non-ionic surface-active agents confer slight protection from loss of strength and improve the crease resistance of the treated varns and fabrics, but have no effect on their elongations.

AUTHORS

Improvement of Tensile Recovery of Vinylon III- Relation between Formalisation and the Tensile Recovery of Vinylon N. Mori and T. Tanaka

J. Soc. Textile Cellulose Ind. Japan,

11, 462-464 (Aug. 1955) When the degree of formalisation exceeds 10 mol. 9 the elastic recovery of vinylon is at first much lowered, and becomes constant at higher degrees of formalisation. Tensile recovery of polyvinyl alcohol fibres obtained by formalisation at lower temperatures and concentrations of H2SO, is not very different from that of ordinary vinylon. The elastic recovery of vinylon is not improved by relaxation in hot water before or after formalisation.

IV—Elastic Recovery of Polyvinyl Alcohol Fibres after Acetalisation with various Aldehydes I. Sakurada, N. Mori, T. Tanaka, K. Sakurai, and

O. Yasutake

Ibid., 526-530 (Sept.)

Polyvinyl alcohol fibres were subjected after heat treatment to acetalisation with formaldehyde (ordinary vinylon), chloroacetaldehyde (vinylon C), acetalyl monosulphide (vinylon 8), benzaldehyde (vinylon B), and dimethylnonal (vinylon N) and their elastic recovery from elongation was compared. In the whole range of elongation vinylons 8 and N show the greatest degree of elasticity. Up to 5% elongation the elasticity of vinylon B also is good, but at higher elongation it is worse than that of ordinary vinylon. In the whole range of elongation vinylon C shows the smallest value of elasticity.

Spraying Loose Wool with Sighting Colours and/or

Pneumatic Conveyors (Huddersfield) A spray nozzle is mounted within a conduit so as to apply atomised liquid to material passing through it, the conduit being so formed that the material is directed on to a surface of the conduit into which sprayed liquid not previously taken up by the material falls.

Metallised Fabrics Deering, Milliken & Co.

BP 741,875 One side of a woven fabric is coated, e.g. by spraying, with a binder containing flake metal. The coating is done to leave the fabric porcus. C.O.C. so as to leave the fabric porous,

Treating Cellulosic Textiles with Polyisocyanates BP 741,137

An anhydrous inorganic salt of a heavy metal, especially a halide of a heavy metal of Group II, e.g. CdI₂ or FeCi₃, is used as the catalyst, being particularly effective when the impregnated fabric is passed through a hot friction

Reducing the Felting Power of Wool by Treatment with Permonosulphuric Acid

Stevenson (Dyers) Treatment with aqueous permonosulphuric acid or a salt thereof at pH <2 or >7 reduces the felting power of wool. If used at pH < 2 then the strength of the acid must be \$10% by weight. Thus all wool fabric (20) is treated for 40 sec. at 33°c. in water (280) containing permonosulphuric acid (4) and sulphuric acid (120) and well rinsed, or similar fabric (30) is treated cold for 30 min. in water (1000), permonosulphuric acid (3) and caustic soda (to bring to pH 10-0). In both cases the treated fabric shows good shrink resistance.

Embossed Pile Fabrics Mohawk Carpet Mills USP 2,700,205 Pile fabric having a pile <15% of which is made of fibres which shrink when heated, e.g. a 70/30 viscose rayonpolyvinyl chloride or a 50/50 polyvinylidene chloridepolyvinyl chloride blend, is embossed in such a manner that only the pressed down pile is heated.

Vinyl Chloride Coated Webs-Leather Substitute

Firestone Tire & Rubber Co. A sheet of vinyl chloride resin is calendered and then a backing of web material, e.g. cloth or paper, pressed against it. The face of the web material pressed against the resin sheet has been previously coated with a flowable unconverted plastisol composition. After the sheet and the web have been united they are heated so that the plastisol is fluxed and converted into a homogeneous layer which is welded to and merged in the resin sheet. C.O.C.

Fused Fabric Assemblies

BP 740,887 Trubenised (Gt. Britain) An interlining for making fused fabric assemblies

contains some fibres which are solid at ordinary temperatures but which become adhesive at a temperature less than the maximum to which the other components can be safely subjected, e.g. polythene. Such interlinings enable fusion of the assembly to be carried out by heat and pressure without presence of plasticisers or solvents.

BP 741,224

The interlining to which a permanent stiff finish is applied contains both thermoplastic and non-thermoplastic components, the latter by themselves forming a coherent factor. The thermoplastic component consists of polythene or other low-m.p. substance.

Heat Treatment of Textiles (I p. 59)

Applying Tension to the Wrappers of Blowing or Decatising Machines (I p. 59)

Coating Webs of Non-absorbent Material with Liquid simultaneously on Both Sides (I p. 59)

Preparation of Polyvinyl Chloride Fibres V— Heat Treatment of the Fibre with Water, Borie Acid, and Formalin. VI—Treatment of the Fibre with Thermosetting Resin (VI p. 69)
Polyvinyl Chloride. XXIII—Some Chemical Treatments

to prevent Thermal Shrinkage of Pure Polyvinyl Chloride (VI p. 69)
Fabrics having a Wool-like Finish (VI p. 70)
Cutting, Dyeing, and Finishing of Velveteen and Corduroy

(VIII p. 71)

1:3:5-Triazines and their Separation by Paper Chromatography (XIV p. 76)

XI-PAPER AND OTHER CELLULOSIC **PRODUCTS**

Increasing the Opacity of Paper by means of Colloidal Hydroxides

J. H. Frasch

XXVIII Congrès International de Chimie Industrielle Chim. et Ind., 74, 171 (Oct. 1955)

The opacity of paper is a function of thickness, porosity, and added pigments. The latter, as inert powders, have no affinity for cellulose, and retention of them is therefore feeble. Certain positively charged colloidal hydroxides, e.g. of ZrO₂, TiO₂, SnO₂, and Al₂O₃, are retained more strongly by the negatively charged cellulose, penetrate better, and have greater covering power than powdered S.R.C.

Water-repellent Finishes on Paper with Methylpolysilazanes N. G. Belen'kaya, M. G. Voronkov, and B. N.Dolgov

N. G. Belen'kaya, M. G. Voronkov, and B. N. Dolgov

J. Appl. Chem. U.S.S.R., 28, 886-898 (Aug. 1955)

Polysilazanes are prepared in ligroin soln. from CH₃.

SiCl₃ and from (CH₃)₂SiCl₃ by treatment with anhydrous ammonia, and the soln. are applied to paper by immersion for various times (the optimum time is found to be 10 min.) and drying at 105-110°c. The water-repellency (approved bydreathin head contact angle imbilities of (supported hydrostatic head, contact angle, imbibition of water after long immersion, etc.) and mechanical properties of the paper are determined. The first of these polysilazanes (polycondensation products of CH_s-Si(NH_s)_s) is particularly recommended for the production of water-repellent finishes and the conferment of improved mechanical and keeping properties on sized and unsized paper. A.E.S.

Effect of an Alkaline Pretreatment on the Hydrolys-ability of Cellulose

V. I. Sharkov and Z. N. Martyshchenko
J. Appl. Chem. U.S.S.R., 28, 881-885 (Aug. 1955)
Pretreatment of cellulose (bleached cotton linters and viscose rayon) with a soln. of caustic alkali (NaOH and KOH) affects the extent of hydrolysis (estimated from the amount of glucose formed) undergone in subsequent treatments at the boil in 10% H_2SO_4 (30 min. and 60 min.). Pretreatments at 18° and 50° c. result in a reduction in the hydrolysability of cotton cellulose when the alkali conen. is low but in greatly increased hydrolysability at higher conen. (12–18% by wt.). When the pretreatment of cotton cellulose is at -18° c. (the cellulose is entered at 0°c., and the mixture is then cooled to -18°c., at which temp. many of the soln. used are frozen), hydrolysability temp. many of the som, used are investing, hydrogeneous increases rapidly as the alkali conen, is increased (for NaOH there is a max. at 4% NaOH). The hydrolysability of viscose rayon is reduced by an alkaline pretreatment, the reduction being at its max. for pretreatments at —18°c. in which the rayon is dissolved completely and then reprecipitated. The structural changes responsible for these phenomena are discussed.

Effect of Sodium and Calcium Ions on the Thermal Stability of Ethylcellulose S. A. Glikman, O. G. Efremova, M. S. Kudryashova, and

A. B. Markman

J. Appl. Chem. U.S.S.R., 28, 877-880 (Aug. 1955) Demineralisation of commercial ethylcellulose by treatment with hydrochloric acid causes a great reduction in thermal stability, but when small amounts of sodium or calcium ions are introduced into the resulting preparation the method already described (see J.S.D.C., 68, 329 1952)) the stability is greatly increased. The effect is attributed to salt formation at carboxyl groups (cf. loc. cit.). A.E.S.

Oxidation of Cellulose. II prepared by a Simple Method S. Machida and M. Inano II- Celluronic Acid

Bull. Chem. Soc. Japan, 28, 343-344 (July 1955) Pulp cellulose is oxidised with sodium nitrite in 90% phosphoric acid to give a carboxylated product. The carboxyl content and the solubility in 1% NaOH soln. increase and the D.P. diminishes with increasing conen. of NaNO₃ and with rise in temp. The carboxyl groups are shown to be almost entirely due to uronic acid residues, the max. value corresponding to 2 uronic residues per 3 glucose residues. D.P. (measured by the viscosity of an acetone soln, of the nitrate esters) decreases from an initial value of ca. 550 to ca. 200. The products with D.P. https://doi.org/10.100/j.new.nitrates.com/ content) are almost completely soluble in NaOH.

Reduction of Aldehyde Groups in Hydrocelluloses by Sodium Borohydride F. S. H. Head

J. Teatile Inst., 46, T 584-T 586 (Sept. 1955) J. Testile Inst., 46. 7 584-7 586 (Sept. 1965)
The reducing power of hydrocelluleses as measured
by Cu number, and their loss in weight en boiling with
dilute alkali, are greatly diminished by treating the
hydrocelluleses with aqueous unbuffered sodium borohydride at 20°c., which shows that this reagent reduces
nearly all the aldehyde groups. There is no effect on the
cuprammonium fluidity. The concentration of borohydride required times are all, with increasing Co. number hydride required rises steeply with increasing Cu number, being 2 M. for the Cu number 4-32. This compares with 0-01 M. borohydride for periodate oxycelluloses of Cu number 3-5, and indicates that the hydrocellulose aldehyde groups are much less easily reduced. Potassium boro-hydride is found to be equally effective. J.W.B.

Hydrolysis of Polysaccharides and other Con-densation Polymers C. M. Stewart and E. J. Williams

Chem. and Ind., 1350-1351 (15 Oct. 1955)
The hydrolysis of "crystalline" polysaccharides seems to be a first-order reaction, while that of "amorphous" polysaccharides seems to follow zero-order kinetics. If the reaction is followed by measuring material dissolved at different times, the fundamental nature of the reaction will be disguised by the physical effect of limiting solubility of chain fragments. Inaccessibility of chains may lead to further complications. Consideration of the distribution of polymer chain lengths leads to the conclusion that the presence of several fractions of very different average D.P. may give rise to apparent sero-order reactions. The mechanisms of hydrolysis of both crystalline and amorphous polysaccharides may be similar. W.R.M.

PATENTS

Paper Sheet for use in making Copies

Standard Register Co. BP 738,512 A sheet of paper, adapted for writing, typing or other inscription, is coated on the reverse side with substantially non-tacky and colouriess material, for transfer at the inscribed areas to another sheet of paper in contact with it. The material contains two reagents, in an acid medium, that react, when the acid is neutralised, to form a darkthat react, when the acid is neutralised, to form a dark-coloured substance. The reagents may be a water-soluble lead, silver, stannous or bismuth salt, and an alkali-metal sulphide, respectively, and the acid medium an acidic manganese salt, e.g. manganese nitrate. The reagents may be associated, in one or two layers, with a water-immiscible volatile organic liquid and a polyvalent metal salt of a higher carboxylic acid, e.g. aluminium, calcium or zinc palmitate or stearate, naphthenate or resinate. One of the reagents may be sodium or potassium sulphide, and bleached shellac may be present to improve the texture of the coating. Dry ammonia gas may be used to cause the transferred coating to become dark and S.V.S.

Water-finished Board Gardner Board & Carton Co. USP 2,698,259 A method of coating paper or board in which the binder is an insolubilised casein and in which water finishing and insolubilising are simultaneous.

Coated Paper for making Planographic Printing Plates 8. D. Warren Co. BP 739,847

Extruded Cellulose Derivatives showing Variegated Effects

Two plasticised thermoplastic organic derivatives of cellulose containing the same substituent but in different proportions are extruded in absence of liquid (except plasticisers) at such a temperature that one is more plastic than the other. This enables attractive effects to be obtained especially if the two cellulose derivatives are differently coloured.

Transfer Paper (IX p. 72)

XII-LEATHER; FURS; OTHER PROTEIN MATERIALS

Structure of Collagen

G. N. Ramachandran and G. Kartha

Nature, 176, 593-595 (24 Sept. 1955) Slight modification of the structural model already proposed by the same authors (ibid., 174, 269 (1954)) results in the correction of certain defects in the earlier model, e.g. lack of agreement with the X-ray data for stretched collegen, and the excessive angle between the NH and N . . . O directions in H bonds. The new structure consists of triple chains of amino acid residues, each chain being itself a helix; the chains form coiled coils, instead of their axes being parallel to the fibre axis. Thus, every third a-C atom is placed upon the surface of a cylinder of radius 1.0 A., successive ones being displaced (in unstretched collagen) by 8.58A. along the axis of the cylinder, and rotated through an angle of 30° about the axis. The single coiled coil repeats itself after 30 residues, and the repeat distance along the fibre axis is 85.8 A. The three chains are symmetrically disposed with respect to each other. A projection of the structure along the fibre axis is given in detail. There is a full discussion of the concordance of the structure with existing X-ray and infrared data. The important rôle of hydroxyproline in collagen, and the periodicity of 040 a. along the fibre axis, may also be explained in terms of the new model, and these points are discussed at length.

J.W.D.

Polypeptide Chain Configuration of Collagen P. M. Cowan, S. McGavin, and A. C. T. North Nature, 176, 1062-1064 (3 Dec. 1955) X-Ray diffraction data suggest a helical structure. A configuration in which three chains, each with three residues per turn relative to the major helix, slowly coil around each other, so that the number of residues per turn, relative to the common axis, is 31, is considered most favourable from analysis of the X-ray diagram. Aminoacid sequence studies do not suggest a rigid sequence, and it is possible that the sequence -prolyl-hydroxy-prolyl-glycyl-, or a similar one, is largely concentrated in one of the three chains, conferring a special configuration on it. The other chains may crystallise about this one. Consideration of the ways in which three poly-L-proline chains can be bonded suggests that the collagen structure consists of three chains of the poly-L-proline type modified to give 31 residues per turn. These may be bound together by NH . . . O hydrogen bonds between residues approximately on the same axial level, and the structure may be further stabilised and the 31 residues per turn maintained by additional hydrogen bonding. X-Ray methods alone may not provide sufficient data to distinguish between different structures of this general type, and the actual structure may be a complex of slightly different arrangements resulting in maximum stability.

New Derivatives of Lanthionine

H. Zahn and F. Osterloh

Annalen, 595, 237-241 (Oct. 1955)

Amide-Iminol Tautomerism

C. A. Grob and B. Fischer

Helv. Chim. Acta, 38, 1794-1802 (Dec. 1955) From ultraviolet and infrared absorption spectra, from bond and resonance energies, and from basic strengths it is concluded that no significant amount of the iminol form is present. PATENT

Tanning with Borated Hydroxymethylphenols American Cyanamid Co. USP 2,701,749

The products obtained by the alkaline condensation of a phenol with formaldehyde and borax are tanning agents which are soluble in water at pH 7-8, yield light-coloured leather and can be used in conjunction with natural or other synthetic tanning agents.

Current Uses of Vegetable Dye Extracts in the Textile Industry (VIII p. 70)

XIII—RUBBER: RESINS: PLASTICS

Plasticisers for Polyvinyl Chloride

Mitt. chem. Forschinst. Wirtschaft Oesterreichs.

XIII-RUBBER; RESINS; etc.

9, (1) 6 (1955)

EPA Technical Digest No. 84

Certain groups are responsible for compatibility with
an plasticising effect on advanced. and plasticising effect on polyvinyl chloride. In order of effectiveness they are (1) carbonic esters, sulphonic esters, phosphoric esters, (2) carboxyl groups, (3) benzene and naphthalene rings and higher condensed rings and their hydrogenated products. Nitro, anino, alcoholic and phenolic hydroxyl, ether groups and halogens have very little effect. The proportion of the active groups present has much more influence than either the polar character of the stabiliser or presence of two carboxyl groups in ester plasticisers. The reaction of polyvinyl chloride to 75 different plasticisers is given in one table while another shows the quantitative influence of the active groups of the plasticisers on their efficiency.

High-energy Irradiation of Polymers- A Literature

S. L. Eislero

PB 111529*, pp. 16 (Nov. 1953) A literature search on the effect of irradiation on the physical properties of polymers. It seems that in some polymers crosslinking can be produced by high energy irradiation and in a variety of elastomers by irradiation with high energy electrons. Natural rubber vulcanisates are improved by neutron radiation but the converse is true for polyisobutylene. Irradiated polyethylene has improved resistance to heat and solvents. COC

Obtainable from T.I.D.U. as microfilm at 4/- or as photocopy at 20/-Stability of Plastics and Elastomers to Nuclear Radiation

C. D. Bopp and O. Sisman

ORNL-1373*, pp. 86 (July 1953) An account of the effects of nuclear radiation on the physical properties of various types and forms of plastics and commercial elastomers. The changes in mechanical properties are correlated with the chemical structure of

the polymers.

Available on loan from T.I.D.U. or photocopies may be purchased from the Science Museum Photocopying Service

PATENTS

Antioxidants for Rubber, etc.

BP 740,155

Compounds of formula-

 $(R^1 = Alk \text{ of } < 16 \text{ C}; R^2 = Alk \text{ or aralkyl of } 2-12 \text{ C};$ both R^1 and R^2 are linked to the benzene nucleus by a primary C atom, e.g. thiobis(3-ethyl-6-a-propylphenol), are powerful antioxidants having little tendency to discolour rubber.

Stabilisers for Polyacrylonitrile Dow Chemical Co.

Addition of 1-20%, preferably 5-15%, by wt. of NN-dimethylhydroxyacetamide, NN-diethylhydroxyacetamide, N-ethylhydroxyacetamide or N-ethyl-N-methylhydroxyacetamide, stabilises polyacrylonitrile against discoloration or becoming brittle upon prolonged exposure to ultraviolet radiation.

BP 739.646

Compounds of formula-

HOC.H.(X)N·CY:O

(X = methyl, ethyl or hydroxyethyl; Y = H or CH_0) e.g. N-(2-hydroxyethyl)-N-methylformamide are similarly

BP 739,650 1-Dimethylaminoethanol, 2-(2-dimethylaminoethoxy)-ethanol, 1-dimethylamino-2-propanol, 3-dimethylamino-1;2-propandiol or 1:3-bis(dimethylamino)-2-propanol are C.O.C. similarly used.

Plasticised Cellulose Esters and Ethers resistant to Light and Heat (VI p. 70)
Transfers for marking Extensible Objects (IX p. 72)

XIV- ANALYSIS: TESTING: APPARATUS

General Method for the Analysis of a Mixture of Two Primary Aromatic Amines differing greatly in Basic Character

A. L. Remizov

J. Appl. Chem. U.S.S.R., 28, 755-760 (July 1955) A mixture of two primary aromatic amines differing sufficiently in basic dissociation const. may be titrated in 0.2 N-HCl with sodium nitrite soln., the less basic amine being preferentially diazotised. The application of the method to mixtures of p-nitroaniline and p-anisidine and of sulphanilamide and aniline is described. A.E.S.

Method of Purifying Direct and Acid Azo Dyes and a New Method of Determining their Purity

B. M. Bogoslovskii

J. Appl. Chem. U.S.S.R., 28, 659-663 (June 1955)
Commercial direct azo dyes are precipitated from aq.
soln. with BaCl., and both direct and acid azo dyes are
precipitated with benzidine hydrochloride. The precipitates are washed free from chloride and then treated with less than the equiv. of sodium carbonate, so that some of the barium or benzidine salt remains undissolved. Filtration and evaporation yield dye of 90-100% purity. The results are as satisfactory as those obtained by the methods of Robinson and Mills (Proc. Roy. Soc., A 131, 576 (1931)) and of Rose (Ind. Eng. Chem., 25, 1028 (1933)), but the method is simpler and more convenient. The purity of an acid or direct search are sent to the second second services. purity of an acid or direct aso dye can be determined by precipitating a weighed amount of dye from soln, with excess of standard benzidine hydrochloride soln, filtering off the precipitate and washing it with water, and titrating the excess of benzidine hydrochloride in the filtrate and washings with standard NaOH in presence of phenol-

1:3:5-Triazines and their Separation by Paper Chromatography

K. Rehnelt

Mh. Chem., 86, 653-661 (Aug. 1955)

The Concept of Swelling Anisotropy. III—Swelling Anisotropy and Elongation on Wetting— Anisotropy and Elongation Applications
J. Pinte, P. Rochas, and J. Henno
J. Pinte, P. Rochas, and J. France,

Bull. Inst. Text. France, (55), 7-22 (Aug. 1955). A simplified method of determining the coefficient of swelling anisotropy C of a fibre is described. The axial swelling $G_{\rm A}$ of the fibre at different regains is measured. and a plot of G_{λ} against regain up to the limit of axial swelling gives a straight line of slope a. The coefficient C is then given by the formula—

$$C = \frac{A}{a} + B$$

where A and B are constants for a given material. A theoretical justification of the formula is given. Measurements on a large number of viscose rayon fibres have shown that the difference in regain at 65% R.H. and at the limit of axial swelling is approximately constant. If A_m is the elongation on wetting of a viscose rayon fibre previously conditioned at 65% R.H., C may be calculated from the formula. from the formula-

$$C = \frac{18 \cdot 6}{A_{\mathrm{m}}} - 0.7$$

involving only a single measurement. An apparatus is described which enables the elongation on wetting of a filament to be measured continuously.

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Research on the Crease Resistance of Cloth. Evaluation of the Effect of the Crease-resistant Finish (X p. 73)

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All inquiries relating to Advertisements in the Journal should be addressed to The General Secretary, The Society of Dyers and Colourists, Dean House, 19 Piccadilly, Bradford 1, Yorkshire.

Replies may be addressed Box —, The Society of Dyers and Colourists, Dean House, 19 Piccapilly, Bradford 1, Yorkshire, where all communications relating to these Advertisements are treated in strict confidence.

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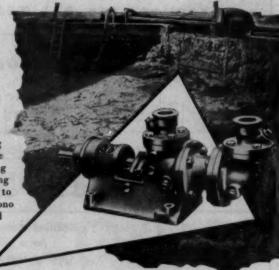
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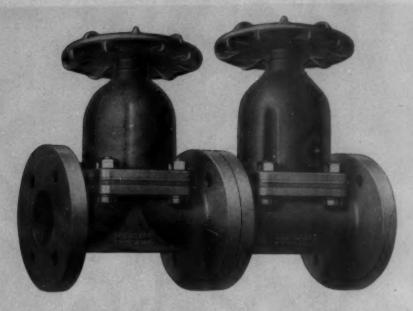




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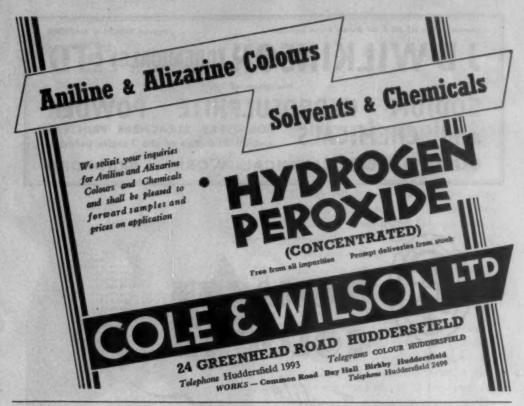
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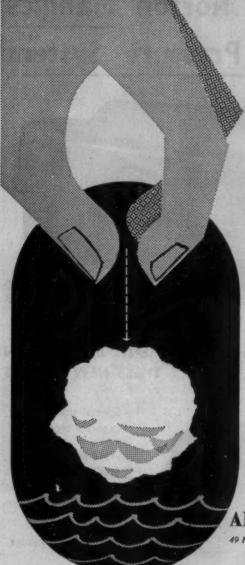
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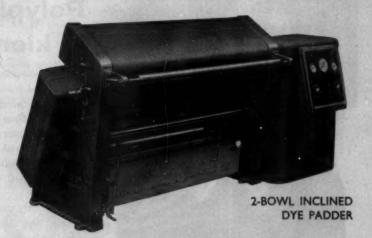
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